

GLASS BONDED CERAMIC BODIES

By

K. N. RAO

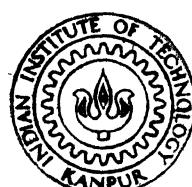
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DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
APRIL, 1985

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A Thesis Submitted
In Partial Fulfilment of the Requirements
for the Degree of

MASTER OF TECHNOLOGY

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By
K. N. RAO

to the

DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
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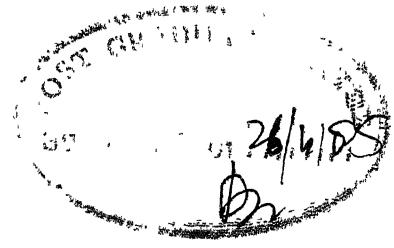
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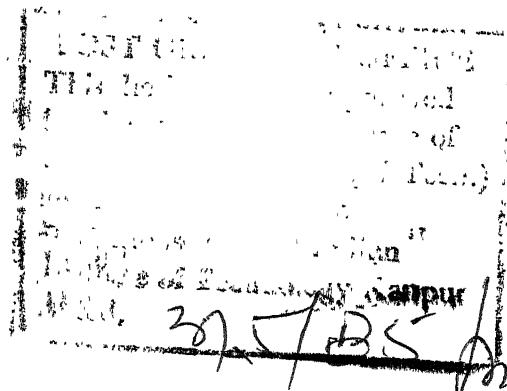
This is to certify that this work entitled, "Glass bonded Ceramic Bodies" by K.N. Rao has been carried out under my supervision and has not been submitted elsewhere for a degree.

Dr. P.C. Kapur
Professor,

Department of Metallurgical Engineering
Indian Institute of Technology, Kanpur

Kanpur

26 April 1985



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ABSTRACT

Studies were conducted on densification of high refractory and hard filler materials embedded in and bonded with preformed glasses. The glasses were available as wastes. The ceramic bodies with Quartz, Fused Alumina, and Thermit Slag as filler materials and three bonding materials, Pyrex glass, Window glass and E-glass, were examined.

Pyrex glass turned out to be the best bonding matrix, producing dense bodies of 95% Theoretical Density and Tensile Strength of upto 200 kg cm^{-2} . A detailed investigation of the process variables shows that there was optimum fineness for filler. The short firing cycle of 8-10 hours and low firing temperatures of $850\text{--}950^\circ\text{C}$ required for glass bonded ceramics could result in significant saving in energy.

CHAPTER-I

INTRODUCTION

In conventional ceramic materials like porcelain, vitreous china, steatite or grinding wheel bodies, the microstructure consists of relatively coarse crystalline phase particles embedded in and bonded by a glassy matrix with different levels of porosity. The properties of the ceramic body depend critically on the nature of the microstructure. The normal firing of these materials is carried out at temperatures of 1200 to 1400°C or even more. These high temperatures are required for the formation of a right quantity of molten phase which is formed by decomposition, melting and reaction between the various components of the batch. The energy consumption for this firing step in the manufacture of these ceramic bodies is high and contributes significantly to the cost.

The present work deals with the fabrication of ceramic materials by an energy-saving low temperature vitrification route. The reduction in densification and vitrification temperatures were achieved by exploiting the low softening temperatures of the commercial glasses, which were used as preformed vitreous phase for the bonding matrix. Use of glass also permitted a much shorter firing cycle as sufficient molten phase, required to bind the crystalline components together was

formed directly by phase change, whereas in conventional bodies considerable time is required to form the pyroplastic mass by chemical reactions between the components.

In this work, three types of waste glasses, Window glass, E-glass and Pyrex glass were used as vitreous bonding matrices and three refractory and hard materials, Quartz, Fused Alumina and Thermit Slag were employed as fillers. The different bond-filler combinations were studied for their densification/vitrification behaviour at relatively lower firing temperatures of 700 to 1000°C.

CHAPTER-II

LITERATURE REVIEW

2.1 Sintering, General Considerations

Here, we use the term 'Sintering' in a generic sense, implying thereby a physico-chemical phenomenon comprising of a series of complex processes which lead to consolidation and bonding of particles together in a ceramic body. The progress of the sintering process can be followed by monitoring changes in the physical properties of the body undergoing sintering, eg., porosity, shrinkage/expansion, strength, etc.

There are three stages of consolidation corresponding to three distinct mechanisms of sintering, (a) Viscous flow of liquid resulting in regrouping and compaction of particles, (b) Solution, followed by precipitation leading to mass transfer and (c) solid phase sintering involving solid phase bulk diffusion, or even vapour phase mass transport^{5,11,12,14}. It should be noted that all the three mechanisms need not operate in a given ceramic system. For example, in the firing of almost pure alumina body, no liquid phase is present and the solid phase sintering is the only mechanism which operates. In many classical ceramic bodies such as porcelains, steatites, cordierites, etc., the first two mechanisms, namely, viscous flow and solution precipitation occur concurrently with very

little solid phase sintering, if at all. Whereas in glass bonded ceramics, where the fillers are high refractory materials, such as those which are described in the present investigation, the viscous flow mechanism predominates. A typical densification curve for sintering in the presence of a liquid phase incorporating all the three mechanisms is shown in fig. 2.1⁵.

2.2 Sintering in presence of liquid⁵

In the presence of liquid, as noted above, two mechanisms can operate. In the first stage when the liquid phase is formed, and if the liquid wets the solid particles, the particles are drawn together by capillary forces with simultaneous infiltration of the liquid into the pores. The net result is a reduction in porosity accompanied by shrinkage of the body. The liquid plays a significant role in rearranging and dense packing of particles in the course of consolidation of the body by reducing the inter particle friction. The shrinkage process by this kind of mechanism can occur fairly rapidly and abruptly at the right firing temperatures, which can lead to serious problems of overfiring and slumping. In case the liquid does not wet the particles, it forms isolated pools : or globes and contributes little or nothing to densification.

In the second stage, not only the fine particles, but also solid material at sharp edges and corners goes into the solution and is reprecipitated elsewhere leading to mass transfer

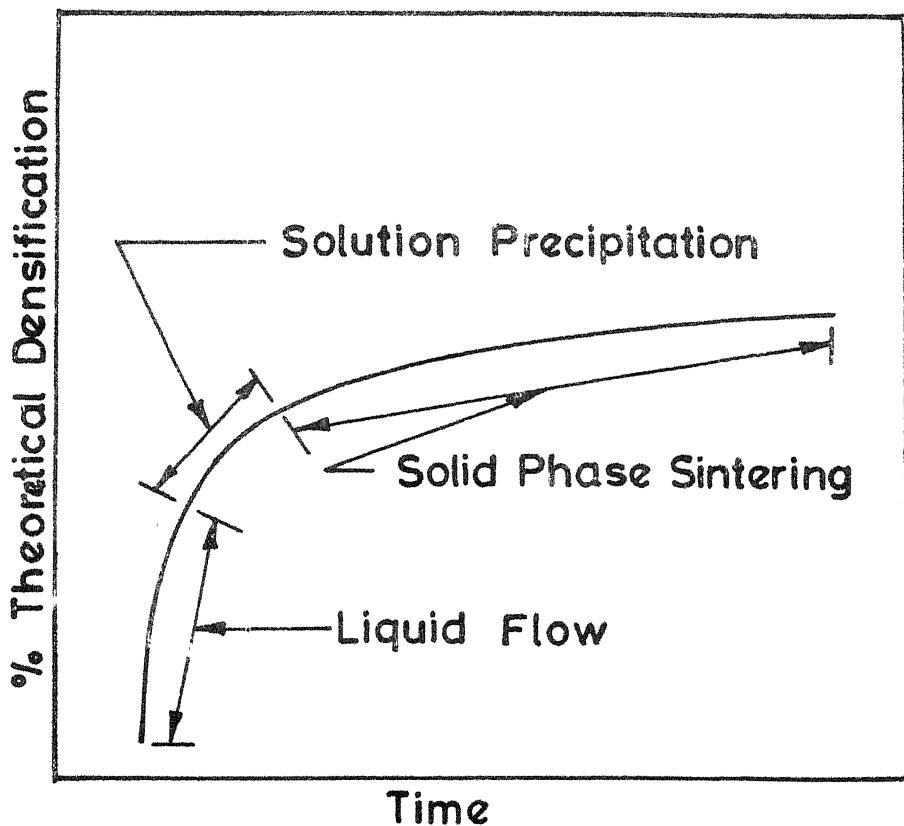


FIG. 2.1 TYPICAL DENSIFICATION CURVE FOR SINTERING IN PRESENCE OF LIQUID PHASE WITH ALL THE THREE MECHANISMS.

and grain growth. This mechanism requires much less liquid than the earlier one, in view of the fact that here the liquid essential function is to provide a conduit for mass transfer as the diffusion rate of species in the liquid phase is substantially higher than in the solid phase. The relative contribution of these two mechanisms is determined by a very complex set of thermodynamic and kinetic considerations and depends on the nature of the system, the phases present and the amount of liquid present.

2.3 Densification and Vitrification by viscous flow mechanism

Since the present work deals primarily with the fabrication of ceramic bodies containing preformed glasses, in which this mechanism predominates, in what follows, the attention will be concentrated on this aspect of sintering. This mechanism of densification and vitrification is most pronounced in those systems where some components are essentially insoluble in the liquid phase formed, at the firing temperature. Such systems are frequently encountered in fabrication of powder metallurgical parts and cermets based on Alumina¹¹.

As mentioned above, this process occurs rapidly once a liquid in sufficient amount having sufficiently low viscosity is formed, resulting in large shrinkages and in general, excellent densification. The net result is a ceramic body having a filler skeleton, embedded in and embounded by a continuous vitreous matrix, with residual pores trapped in the microstructure.

The volume of the pores in the green compact lies in the range of 25 to 50 percent depending on the particle size distribution, their packing and the moulding pressure employed. With sufficient quantity of liquid phase, it is possible to attain the theoretical density by the viscous flow mechanism only which essentially entails regrouping, rearrangement and packing of particles and filling up of the pores by the liquid. According to Cannon and Lenel¹², the quantity of liquid must be at least 25% by volume, on the other hand, Kingery⁷ has calculated that, for spherical filler particles, the minimum quantity of liquid phase for complete densification is 35% by volume. It should be noted that this number is meaningful if the spheres are of single size and pack densely, because only then the packed bed has a porosity of 35%. In fact, Sremenko, et al.⁵, have found that 50% liquid phase is necessary for attaining the theoretical density in real systems. This is understandable because in real systems particles are not only angular and possibly asymmetric, but are also distributed in size and shape.

In presence of insufficient liquid phase, other sintering mechanisms must be superimposed in order to attain complete densification. As said by Kingery⁹, given adequate amount of liquid phase, it is possible to attain complete densification

by viscous flow only and without any change in the grain shape in suitable conditions provided the filler materials exhibit no meaningful solubility in the liquid at the firing temperature.

2.4 Effect of particle size

Kingery⁷ has shown that the degree of vitrification by viscous flow mechanism is inversely proportional to the particle size, whereas in the solution reprecipitation mechanism, it is inversely proportional to the radius to the power 4/3.

Linel¹² found that the particle size of the filler is a critical parameter in this type of densification mechanism. In Tungsten - Copper system studied by him the extent of vitrification fell markedly when Tungsten powders coarser than 3-5 microns were used. On the other hand with fine powders, although density of green compacts was less, on firing the samples showed greater vitrification and superior mechanical properties. It is hypothesized that the disadvantages of the large size particles are the following, (i) Larger the filler size, greater would be the stresses set up due to thermal expansion/contraction mismatch between the filler and the bonding matrix, (ii) In case of fine size filler, the bonding contact area will be substantially higher, (iii) The apparent viscosity of the pyroplastic mass, mixture of molten phase and the particulate solid, would be significantly higher for the fine size fillers, thereby bestowing considerable mechanical stability to the body.

2.5 Effect of Compacting Pressure

It is well known that fine particles desirable for densification are more difficult to compact mechanically. Therefore high compaction pressures are in general necessary. Plate¹³ noted that the compacting pressure not only reduces the firing shrinkages but also the temperatures of initial interaction between the components. In general higher the compaction pressure, less is the volume of pores that need to be illuminated in vitrification, therefore less is the shrinkage and greater is the dimensional accuracy of the piece.

On the other hand, high compaction pressure can lead to the formation of narrow-necked capillaries or closed pores, due to fracture of particles into ultrafine fragments and even elastic deformation. In firing, considerable gas pressure can build up in these closed pores. As a consequence, this pressure counteracts the capillary pressure and may reduce the rate of flow of liquid into the pores, thereby slowing down the rate of densification. In case the gas pressure exceeds the capillary pressure, the sample may even exhibit expansion or even bloating. In summary, higher compacting pressure has a beneficial effect on sintering provided extensive formation of closed pores is avoided.

2.6 Densification of Glass Powder

The viscous flow mechanism operates when a compact of

powdered glass is sintered as shown by Frenkel³ and, Kuczynski and Zapalatynski⁴. The contribution of mass transfer by the diffusion mechanism seems to be insignificant.

Cutler and Henrichsen² found that crushed glass sinters much more rapidly than spherical glass. The rate of shrinkage depends not only on viscosity but is also inversely proportional to the radius of the particle, because the rate at which the glass softens enough for the flow to occur is inversely proportional to the particle size. And for the same reason, crushed glass particles which have numerous sharp corners or edges would also soften more readily than smooth spherical particles. These authors infact found that the rate of densification of crushed glass was five times that of spherical glass. Hence the particle shape plays an important roll in viscous flow mechanism of vitrification.

2.7 Glass-Filler Bodies

In sintering of porcelain, vitreous china, cordierite temperatures of 1200-1400°C or above are needed in order to obtain dense and nominally non-porous products. The reason for these high temperatures is that the components in the raw batch mix must react to form a certain amount of molten phase required for densification. The formation of the molten phase occurs usually above 1050°C. It then seems logical to formulate ceramic bodies containing a preformed vitreous phase in a form of powdered

glass in which case the firing temperature and time should be significantly less. Not only this, the glassy components of the raw mix are available as municipal wastes or industrial wastes in sufficiently large quantities.

In a major contribution to this field Wiegmann¹ prepared dense ceramic electrical insulation materials at less than 1000°C. According to him the requirements for successful fabrication of glass-filler type ceramics are the following.

1. The crystalline or filler phase content must be as high as possible in order to assure the maximum strength.
2. For the same reason, the particle size of the filler should be as small as possible consistent with the energy expenditure and contamination in grinding.
3. The fired body understandably should have minimum number of closed pores, although it is not clear how one should go out assuring the absence of these pores except by taking intuitive measures such as particle size control and limit on compaction pressure.
4. Minimise thermal stresses between the glassy matrix and the crystalline phase.
5. Employ glass which softens sufficiently below 1000°C in order to achieve densification at relatively lower temperatures.

As a matter of fact Wiegmann found that the temperature needed to achieve a certain degree of densification increased with increase in the softening point of glass as well as with increase in filler quantity and its particle size. In agreement with the work of Cutler and Henrichsen², he also found that firing temperatures are lower if the particle size of the glass is finer.

Wiegmann claims that in firing filler-glass bodies at 850-1000°C, the energy consumption is of the order of 3 to 6 MJ/k as against 16-25 MJ/kg product needed in conventional fabrication route. Another advantage claimed is the much larger life of the refractory lining in the kiln operating at lower temperatures, as well as higher output due to shorter cycle.

CHAPTER-III

OBJECTIVE OF PRESENT WORK

The filler-glass ceramic bodies seem to be an attractive route for producing a host of ceramic materials at low temperatures in which at least one component, namely glass is a waste material available in large quantity quite cheaply. The central aim of this work was to demonstrate the feasibility of producing glass bonded ceramics at temperatures less than 1000°C using the waste glass available in India for example, ordinary Window glass, Pyrex glass and E-glass, in combination with three refractory fillers Quartz, Alumina and another waste product Thermit Slag.

The objective was

1. To study the effect of the following process parameters on the Quartz/Alumina/Thermit Slag based filler-glass ceramic bodies :
 - i) Type of glass
 - ii) Glass-filler ratio
 - iii) Fineness of the filler
 - iv) Compaction pressure
 - v) Firing temperature

2. To finalise the optimum composition and processing parameters for each of the three fillers from the view point of different applications where the extent of densification varies with products.

CHAPTER-IV

MATERIALS, METHODS AND TESTS

4.1 Materials

The raw materials used were Window Glass (WG), Pyrex Glass (PG), E-glass (EG), Quartz (Q), Alumina (A) and Thermit Slag (T).

4.1.1 Glass

All the glasses used were waste materials. Window glass is a soda lime silicate glass, having relatively low softening points. Pyrex glass is the brand name used in the manufacture of laboratory ware. This is a low expansion boro-silicate glass. E-glass is a type of low alkali lime-alumina-borosilicate glass, used in manufacture of glass fibres. All glasses were ground to fine size before use. The chemical composition and other properties of these glasses are given in the Table 4.1, and the viscosity-temperature behaviour is given in the figure 4.1.

4.1.2 Alumina, Quartz and Thermit Slag

White Fused Alumina (+ 99% pure) and Ceramic Quartz, both of -100 mesh size were taken and ground for different times. Quartz occurs in a number of polymorphs. The principal transformations are as follows :

Table 4.1 : Chemical Composition and Properties of glasses

Property	Window glass	Pyrex glass	E-glass
Chemical (wt%)			
SiO ₂	72.0	80.5	54.5
B ₂ O ₃	-	12.9	8.5
Al ₂ O ₃	1.0	2.2	14.5
CaO	11.0	-	22.0
MgO	2.5	-	-
Na ₂ O	13.0	3.8	0.5
Others	0.5	0.6	-
Strain Point °C	515	520	-
Anneal Point °C	550	565	-
Softening Point °C	730	820	830
True density gm cm ⁻³	2.472	2.252	2.555
Coefficient of Thermal expansion x 10 ⁷ per °C	85	32	60

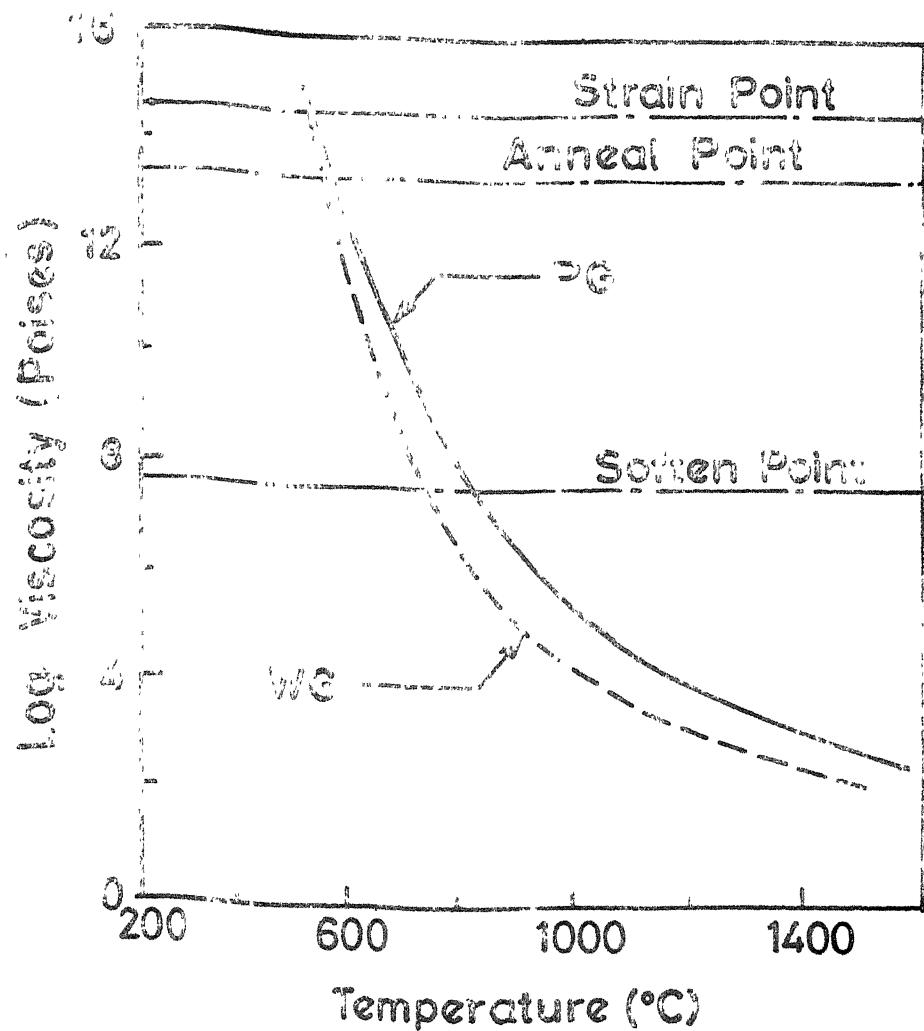
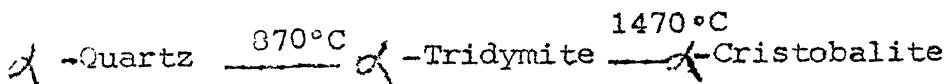


FIG. 4.1 VISCOSITY CURVES OF GLASSES.



In addition, there is a reversible inversion between β -Quartz and α -Quartz at 573°C . Eventhough the experiments in this work were conducted at below 1000°C , partial transformation of Quartz to higher polymorphs cannot be excluded.

Thermit slag was obtained in lumps from N/S ABM Metalloys Ltd., Shimoga, Karnataka. This was crushed and ground to ~ 100 mesh size for further processing. Its chemical composition in table 4.2 shows that it is a high Alumina waste material with substantial amounts of MnO and CaO.

Table 4.2 : Chemical Composition of Thermit Slag (wt%)

SiO_2	2.10	CaO	6.00
Al_2O_3	80.30	MgO	0.90
MnO	10.1	FeO	0.16

The true densities of the three filler materials determined by Pyknometer method are as follows :

Alumina	3.98 gm cm^{-3}
Quartz	2.68 gm cm^{-3}
Thermit Slag	3.214 gm cm^{-3}

4.2 Methods

4.2.1 Fine Milling

All the raw materials were fine ground separately. The waste glasses were washed clean and first broken to -5mm size in an ordinary cast iron mortar and pestle. The crushed glass was cleaned again and then wet ground in a ball mill. The filler materials being already-100 mesh size, were wet ground directly. The glasses were ground for 25 hours, and the three filler materials for 10, 20 and 40 hours. The specifications of the ball mills were as follows :

Ball mill for grinding filler

Height	120 mm
Internal diameter	121 mm.
External diameter	143 mm
Useful volume	1.14 lit
Revolutions per minute	115
Weight of filler taken	250 gm
Weight of high alumina grinding media of size 12.5 x 12.5 mm cylinders	1 kg

Ball mill for grinding glass

Height	200 mm
Internal diameter	210 mm
External diameter	232 mm

Useful volume	6 lit
Revolutions per minute	72
weight of high alumina grinding media of size	
20x20 mm cylinders	2.25 kg
12.5x12.5 mm cylinders	2.25 kg
Weight of glass taken	1.50 kg
The quantity of water varied with different materials.	
225 ml per 250 gm of Quartz taken	
250 ml per 250 gm of Alumina taken	
400 ml per 250 gm of Thermit Slag taken	
2 l per 1.5 kg of glass taken.	

4.2.2 Pyrometric Cone Tests of Filler-Glass Compositions

For preliminary screening and to obtain a qualitative idea of the softening and flow behaviour of various filler-glass compositions, these mixtures were made into standard laboratory size cones and subjected to pyrometric cone tests, i.e. the temperatures at which the cones initially tipped, half tipped and fully tipped were noted.

Cone making and mounting : Ground filler and glass in different proportions were thoroughly mixed and 4% dextrin was added. The wet mix was moulded into the shape of cones in a steel mold and dried in an oven at 80°C. Lower percentage of dextrin did not provide enough green strength whereas higher dextrin contents lead to cracking of the cones in firing. These dry cones were mounted on a circular alumina plate called plaque which had

suitable grooves cut in the periphery for the purpose. Refractory clay was used to cement the cones in place. An inward slant of about 10° to the vertical was maintained for all the cones.

Size of the cones

1. Overall height (between planes perpendicular to the triagonal axis) = 28.56 mm
2. Length of the sides of the triagonal tip = 1.91 mm
3. Length of the sides of the base = 7.14 mm

Tipping of cones : The plaque with the cones was heated uniformly at a rate of 2°C min and the temperatures at which the cones started softening i.e. began to tip, bent one half and fully tipped were noted down by means of a Pt-Rh thermocouple placed in the centre of the plaque.

4.2.3 Pellet making :

Small Pellets : Pellets of diameter 12.5 mm and thickness ranging from 3 to 8 mm were made in a three-piece split die. Two drops (1 drop $\approx 0.05 \text{ cm}^3$) of 5% dextrin solution was added to each gram of dry powder mix, for providing adequate green strength to the pressed pellets. After thoroughly mixing the filler and glass powders in a porcelain mortar and pestle, the mixture was moulded in a hydraulic press at any of the two levels of pressures, 375 kg cm^{-2} or 1500 kg cm^{-2} . Adequate cleaning of die with Acetone and lubrication with Liquid Parafin was

practised. The pellets were dried for more than a day at around 100°C before firing. These pellets were used to monitor the densification of the body.

Big pellets : For strength testing, bigger samples of 25 mm diameter and 8 to 12 mm thick were pressed. Now dextrin was not used as to permit a faster rate of firing. Only a few drops of water were enough to give adequate green strength for handling after pressing at 1500 kg cm⁻². The big samples were thoroughly dried for 30 hours at 100°C. As some surface cracks showed up after drying in many samples, these were removed by carefully grinding the dry green pieces on a belt grinding machine so as to remove about 2 mm of the thickness. Each sample was marked, its bulk density was determined and then fired.

4.2.4 Firing

Pellets were fired in a tube furnace of diameter 5 cm. The pellets were loaded one high on a bed of coarse alumina particles. For big pellets a heating rate of 5°C min⁻¹ was used. On account of the presence of dextrin in small pellets, slower heating rate of about 3°C min⁻¹ was adapted for these. Samples containing quartz were also heated at a slower rate specially in the 550-600°C range where β -Quartz to α -Quartz transformation occurs. The pellets were soaked at the maximum temperature for one hour for small and two hours for big pellets, after which they were furnace cooled to room temperature. Typical firing

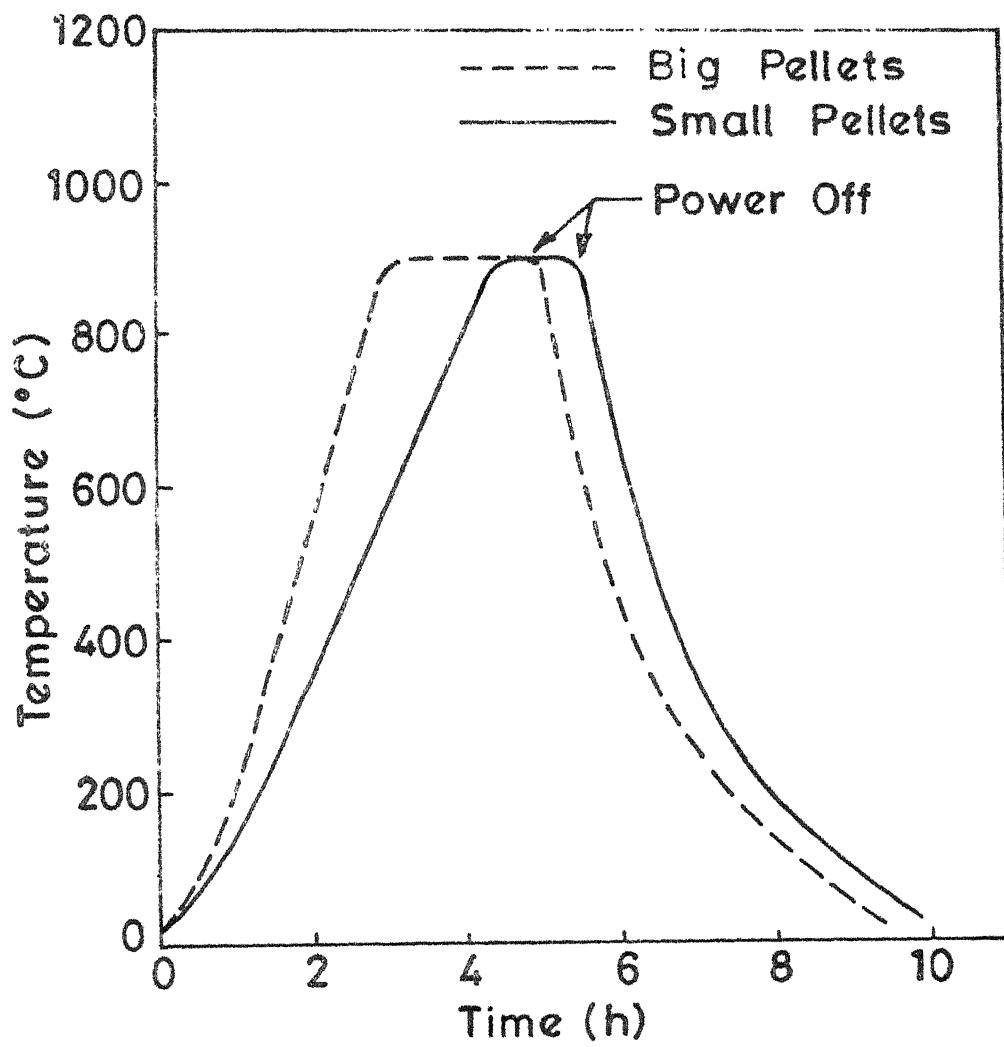


FIG. 4.2. FIRING CYCLES FOR PELLETS.

curves are shown in fig. 4.2. Same set of small pellets was used to get densification data at different firing temperatures, i.e. 700, 750, 800, 850, 900, 950 and 1000°C.

4.3 Tests

4.3.1 True Density of Powders

True densities were determined by the pyknometer method. Water was used as the displacement liquid. Care was taken to remove any air bubbles trapped in the powder. The pyknometer bottle with the powder and half filled with water was maintained at 100°C in a boiling water bath for around 10 minutes so that the entrapped air bubbles were driven out. The weight measurements were taken after the pyknometer and its contents cooled down to room temperature. Data on the densities of all the materials are given in Appendix I.

4.3.2 Bulk Density of Pellets

Bulk densities of small pellets were measured using the mass and volume of the pieces; the latter was measured by micrometer. Bulk densities of big pellets were measured by water displacement. The sample was weighed in air, boiled in water for half an hour, weighed under water, and then weighed while still soaked with water, in air.

4.3.3 Surface Area of Powders

Specific surface areas of all the powders were determined using Blaine's apparatus and a standard sample of Portland Cement.

The method involves preparation of a uniformly packed bed of known porosity (usually 50%) in a standard cell and noting the time taken for a fixed volume of air at constant temperature to pass through this bed.

The formula is

$$S = S_s \frac{\rho_s (1-e_s)}{\rho (1-e)} \sqrt{\frac{e^3}{e_s^3} \cdot \frac{\eta_s}{\eta}} \sqrt{\frac{T}{T_s}}$$

where,

S = Specific surface area in $\text{cm}^2 \text{ gm}^{-1}$ of the test sample.

S_s = Specific surface area in $\text{cm}^2 \text{ gm}^{-1}$ of standard sample used in calibration of the apparatus. Here ordinary Portland Cement was used ($S_s = 2250 \text{ cm}^2 \text{ gm}^{-1}$)

T = Measured time interval in seconds of manometer drop for test sample.

T_s = Measured time interval in seconds of manometer drop for sample used in the calibration of apparatus.

η = Viscosity of air in poise at the temperature of test of the test sample.

η_s = Viscosity of air in poise at the temperature of test of the standard sample used in calibration of apparatus.

e = Porosity of prepared bed of test sample

e_s = Porosity of prepared bed of standard sample (Portland Cement)

ρ = True density of test sample in gm cm^{-3}
 ρ_s = True density of standard sample used in calibration
 of apparatus (Assumed to be 3.15 gm cm^{-3} for
 Portland Cement)

When the measurements are done at the same temperature, the formula simplifies to

$$S = S_s \frac{\rho_s}{\rho} \frac{(1-e_s)}{(1-e)} \sqrt{\frac{e^3}{e_s^3}} \cdot \sqrt{\frac{T}{T_s}}$$

Specific surface area values of all the materials are given in the Appendix I.

4.3.4 Splitting test :

The big disc shaped pellet was placed between the platens of the Instron testing machine such that its axis was horizontal as shown in the figure 4.3. The load was increased at a rate of 0.05 mm min^{-1} cross head speed until failure occurred by splitting along the vertical diameter of the disc. When the load is applied along the generatrix, then an element near the end of vertical diameter of the cylinder is subjected to a compressive stress of

$$\frac{2P}{\pi HD} \left[\frac{D^2}{r(D-r)} - 1 \right]$$

and a horizontal tensile stress of

$$\frac{2P}{\pi HD}$$

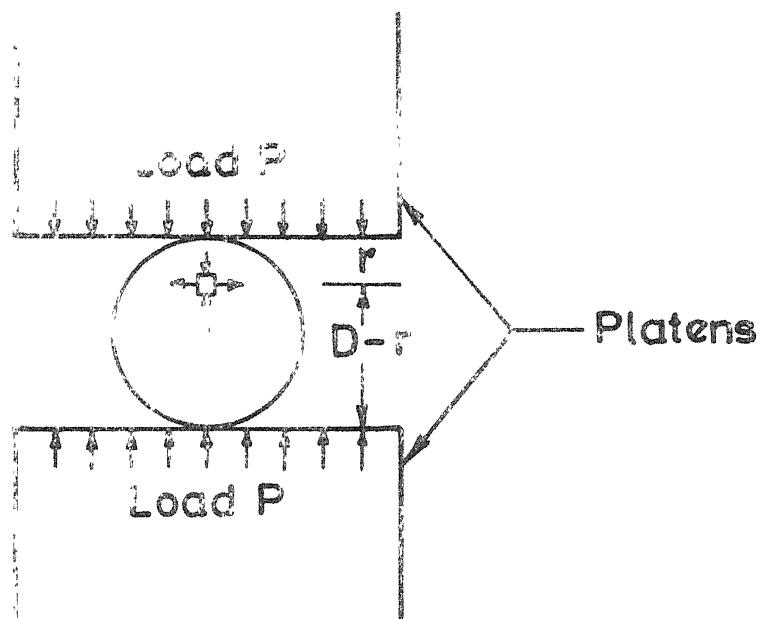


FIG. 4.3. CONFIGURATION OF SPLITTING TEST.

where P is the compressive load on the disc

H is the height of the disc

D is the diameter of the disc

The splitting occurs due to this tensile stress and hence the tensile strength of the sample can be calculated from above.

CHAPTER-V

EXPERIMENTAL RESULTS

5.1 Grinding of Raw Materials

The filler materials were ground for 10, 20, 40 hours and the glasses for 25 hours as described in section 4.2.1. The specific surface areas of the powders as a function of grinding time, are shown in figure 5.1. It can be seen that, as expected, increment in fineness decreases as the time of grinding increases because the energy required for grinding the materials becomes disproportionately high as the particles become finer.

5.2 Preliminary Experiments

As the aim was to produce ceramic materials at relatively low firing temperatures, the first experiments were conducted to estimate the probable vitrification temperature range, by the pyrometric cone tests. The tipping temperatures of test cones subjected to a constant rate of heating are shown in figures 5.2 and 5.3.

The first figure shows Window Glass-Quartz compositions in which the data is exhibited in three separate boxes for 10 h, 20 h, and 40 h ground Quartz. Each box has three curves for initial tipping, half bent and fully tipped temperatures as a function of percentage bond by volume. This data is plotted again in the second figure in which, each box pertains

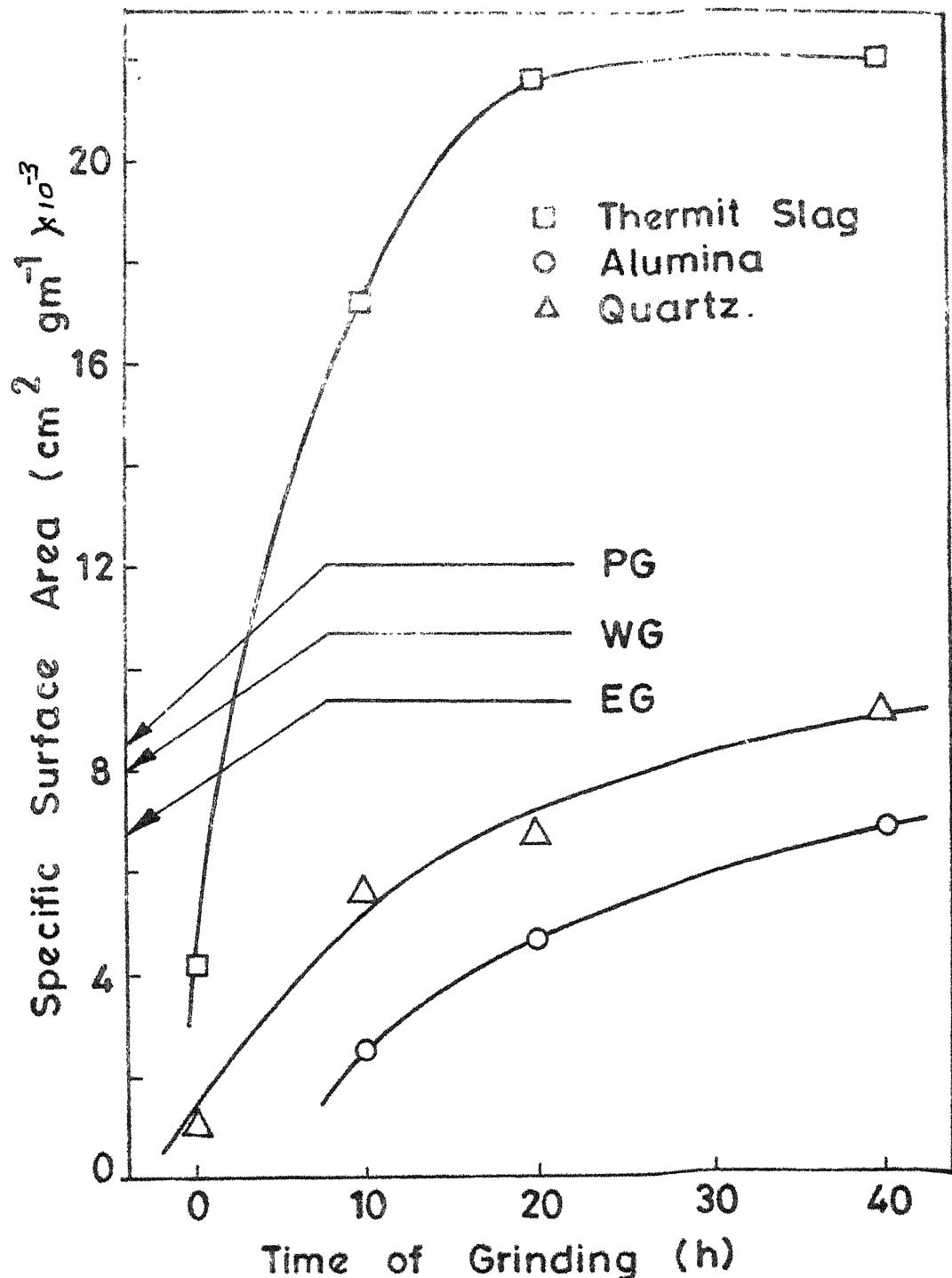


FIG. 5.1. INCREASE IN SPECIFIC SURFACE AREA WITH GRINDING TIME.

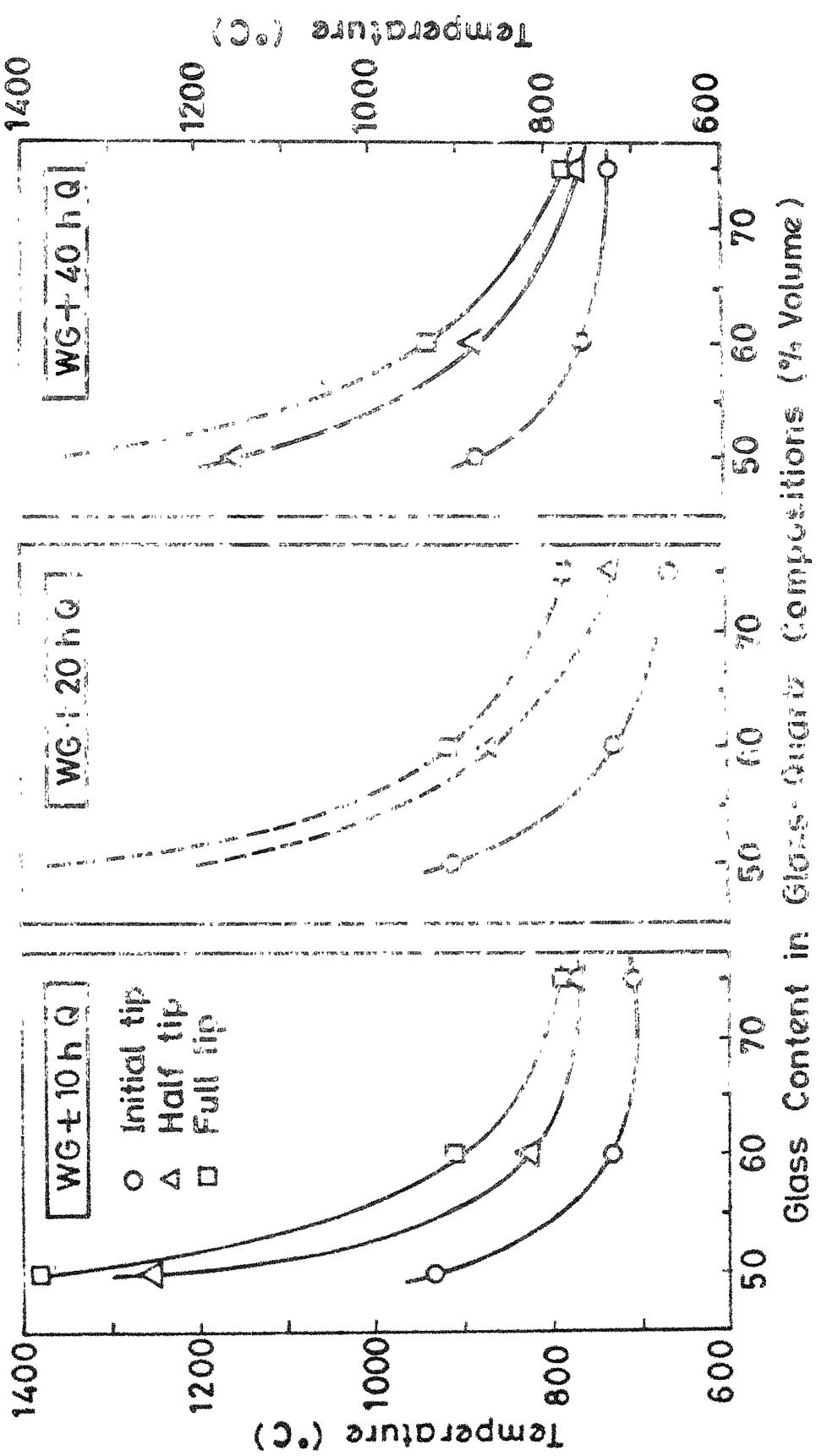
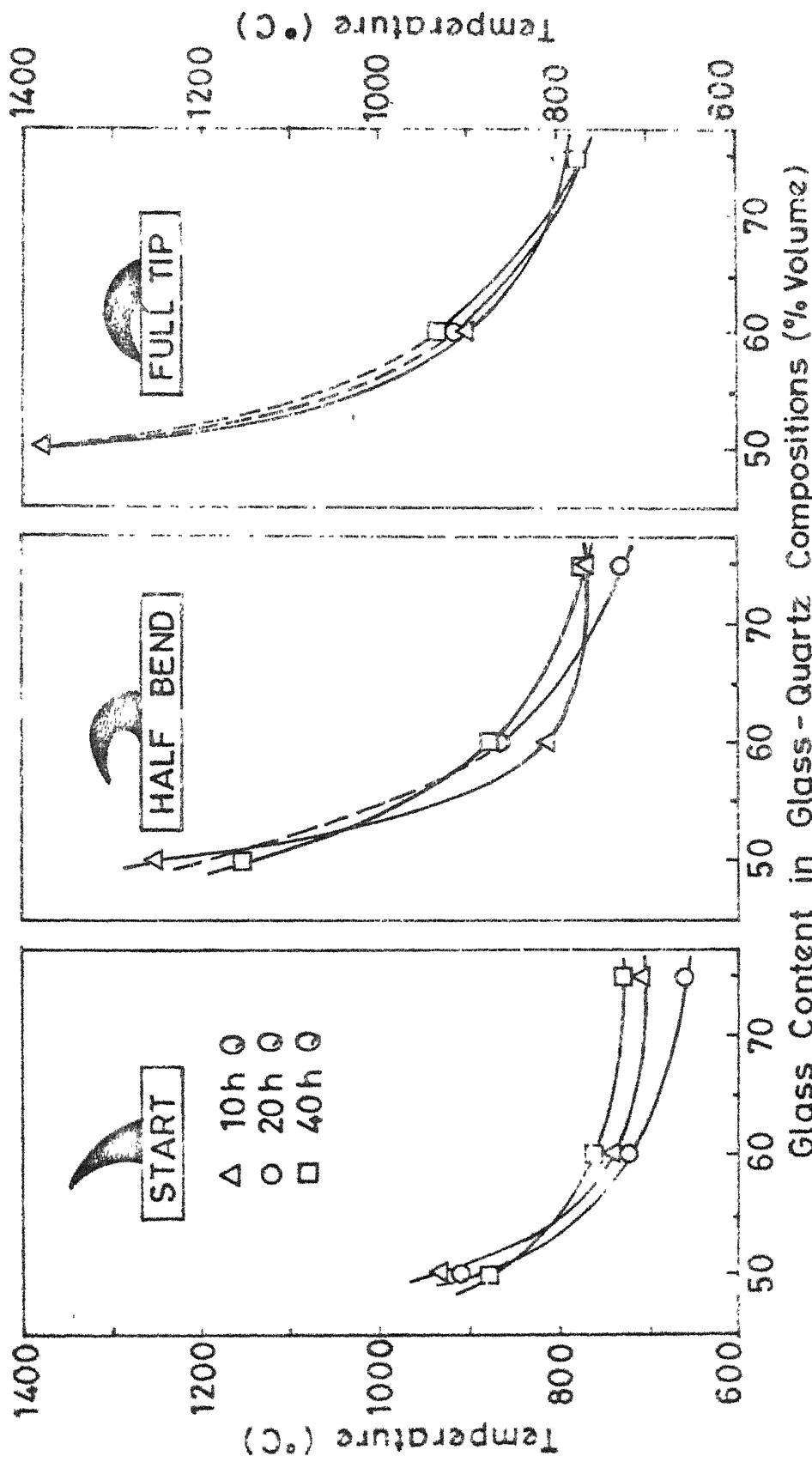


FIG. 5.2. TEMPERATURES OF INITIAL TIP, HALF TIP, AND FULL TIP OF PYROMETRIC TEST CONES MADE OF DIFFERENT WINDOW GLASS-QUARTZ COMPOSITIONS.



to the state of cone namely initial tipping, half tip and fully bent.

It will be noted that in general between 50% to 60% bond by volume, there occurs a sharp drop in temperature for all three states of cone tipping. Between 60 and 75% bond by volume, the drop in temperature is not so sharp. It also seems that for the temperatures of fully tipped cones the fineness of Quartz is not a matter. But there are significant differences for the temperatures corresponding to initial and half tipping. Comparison with latter experiments on pellets lead one to believe that temperatures in the vicinity of initial tipping are the required firing temperatures for maximum densification attainable.

In view of large amount of time needed for carrying out the pyrometric cone test, it was decided at this stage to proceed directly to the pressed pellets. Further it was decided to use the finest powders available and hence further experiments were conducted with only 40 h ground Quartz, Alumina and Thermit Slag.

5.3 Selection of Vitreous Bonding Matrix

Each filler was bonded with three types of glasses, in 60 and 50% volume of glass. Thus in all 18 compositions regulated. Pellets of 1.25 cm diameter were pressed under 375 kg cm^{-2} pressure and fired in the range of 700 to 1000°C . The results are exhibited by figures 5.4, 5.5 and 5.6 for Quartz, Alumina and

Slag respectively.

Figure 5.4, shows the percentage of theoretical density (% TD) attained as a function of firing temperature for 6 compositions in which three glasses at 60% and 50% volume levels each were used. It will be seen that 60% Pyrex Glass and 50% Window glass, gave the best resulted. With 60% Window glass, the samples showed very poor densification. In general, as expected, higher the percentage of glass content, higher was the relative theoretical density attained at a given firing temperature.

Figure 5.5 for Alumina similarly shows the marked superiority of 60% volume of Pyrex glass over all other bonds, resulting in a densification of 92% of theoretical density at 1000°C. Interestingly enough with Window glass at both 50% and 60% levels, the percentage theoretical density attained is invariant of temperature in the range of 800-1000°C. Another note worthy feature is the very large difference in the densification results between 60% and 50% bonds for Pyrex glass. Again the E-glass showed disappointing results as far as densification is concerned.

Figure 5.6 for Thermit Slag again shows the superiority of Pyrex glass at 60% levels over other bonds, plus the fact that the maximum relative theoretical density reached is invariant of the firing temperature over the range 850 to 950°C.

To summarise these results, under the experimental conditions obtaining, Pyrex glass is by far the best bonding matrix.

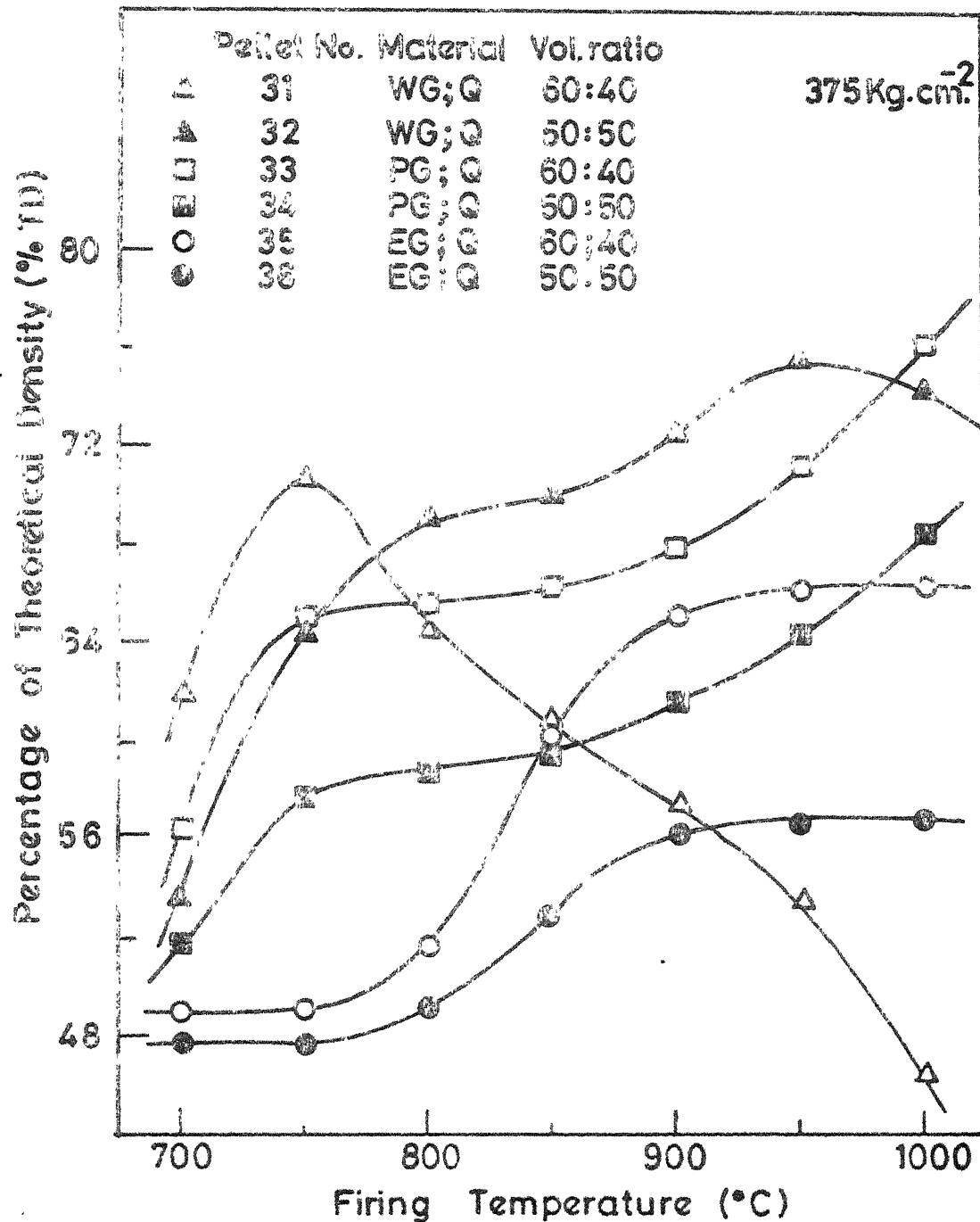


FIG. 5.4. PERCENTAGE OF THEORETICAL DENSITY ATTAINED AT DIFFERENT FIRING TEMPERATURES FOR 40h GROUND QUARTZ BONDED WITH 3 GLASSES.

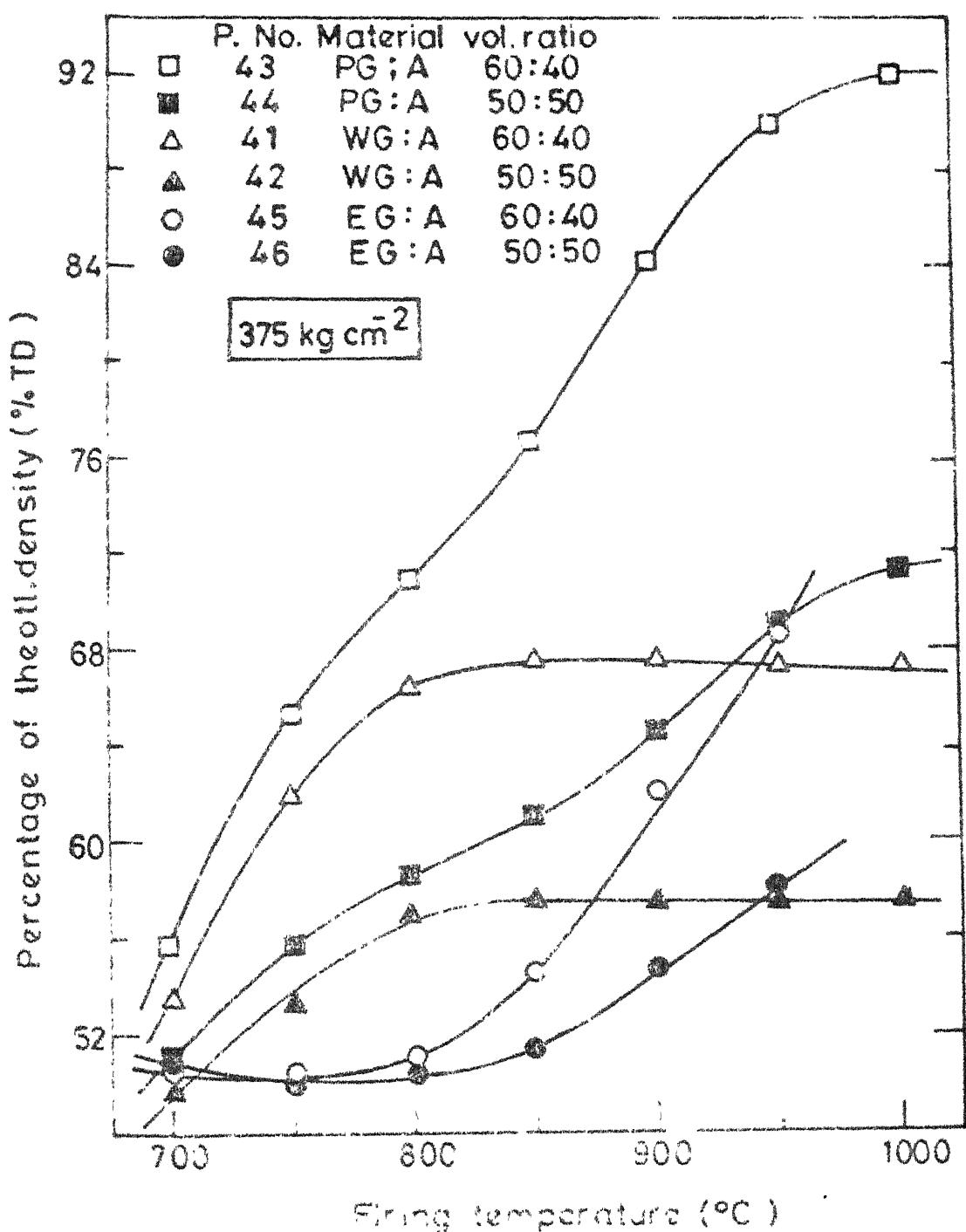


FIG 5.5 PERCENTAGE OF THEORETICAL DENSITY ATTAINED AT DIFFERENT FIRING TEMPERATURES FOR 40% GROUND ALUMINA BONDED WITH GLASSES

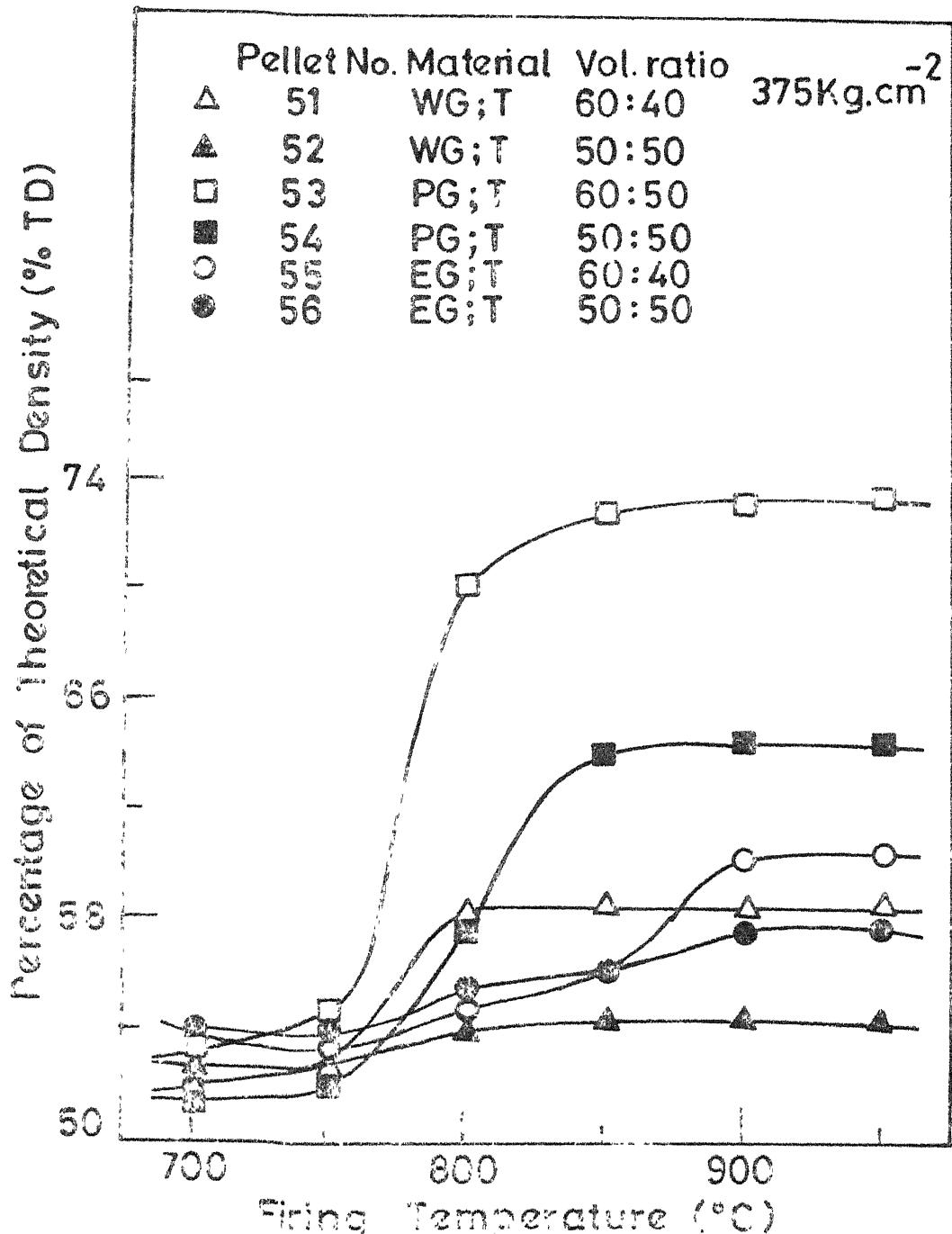


FIG. 5-6. PERCENTAGE OF THEORETICAL DENSITY ATTAINED AT DIFFERENT FIRING TEMP. FOR 40% GPG AND SLAG BONDED WITH 3 GLASSSES

It is possible to attain densification of 90% plus, for Alumina and about 75% for Quartz and Thermit Slag with 60% volume of Pyrex glass.

5.4 Enhancement of Densification

From the above results, for two fillers Quartz and Thermit Slag, the densification achieved was only of the order of 75% and needed to be enhanced further. For this purpose two changes were made i) increase in the moulding pressure from 375 kg cm^{-2} to 1500 kg cm^{-2} and ii) increase in Pyrex glass bond content from 60 to 65 and then to 70% by volume. The results are shown in figures 5.7, 5.8 and 5.9 for Quartz, Alumina and Thermit Slag respectively, along with earlier results with 60% volume bond and moulding pressure of 375 kg cm^{-2} .

Fig. 5.7 shows, as expected, that the densification improved with increase in moulding pressure as well as with higher bond content and with 1500 kg cm^{-2} moulding pressure, it was possible to attain 90% of theoretical density. This calculation is done on the assumption that the Silica remains in the form of Quartz with density of 2.68 gm cm^{-3} . It is however possible that with the thermal treatment done, some quartz has undergone transformation to higher polymorphs, Tridymite and Cristobalite which have significantly lower densities. If this indeed had happened, the achieved densification obtained would be somewhat higher, perhaps as high as 95%.

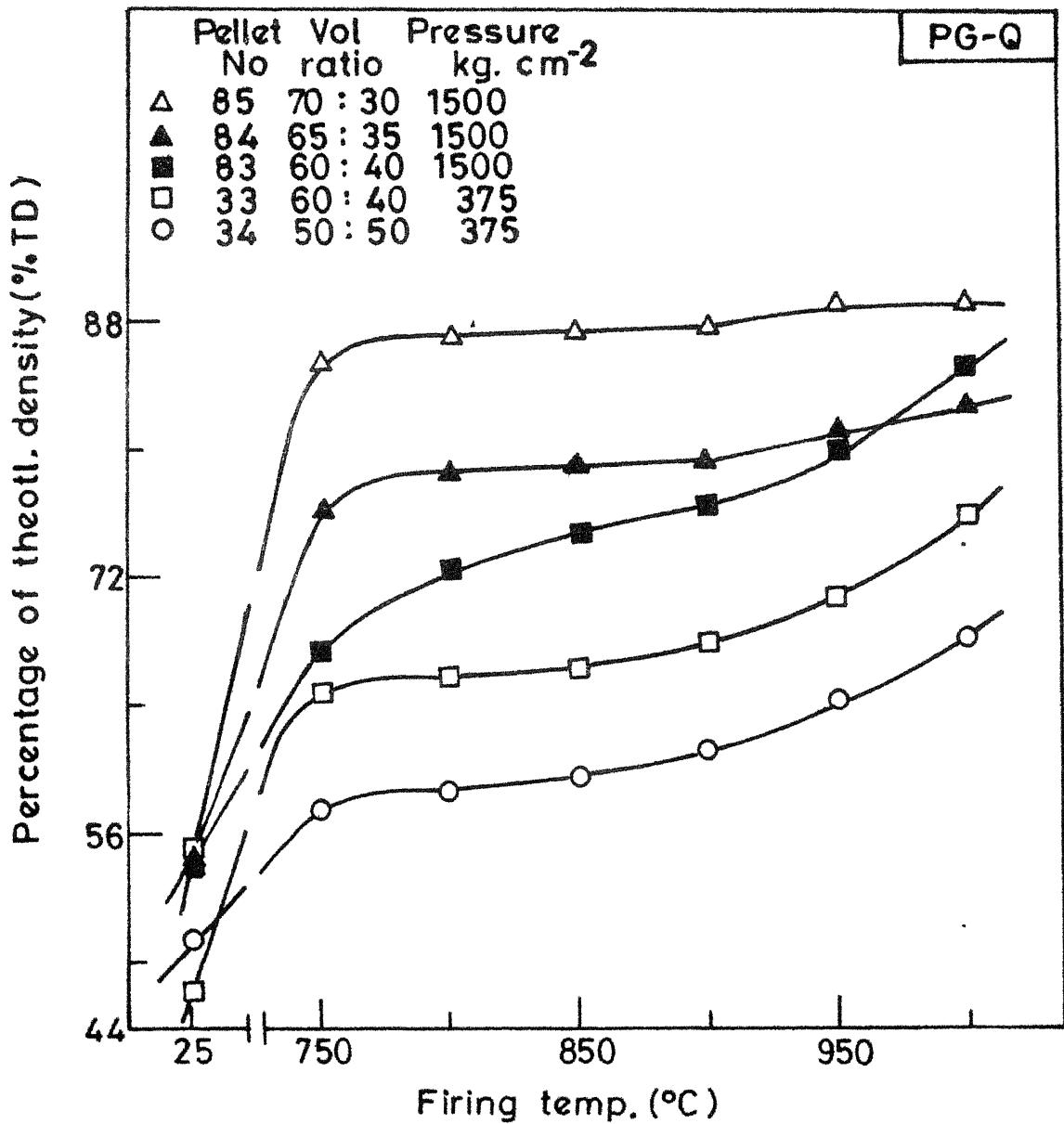


FIG. 5.7 PERCENTAGE OF THEORETICAL DENSITY AS A FUNCTION OF FIRING TEMPERATURE FOR PYREX - QUARTZ (40h) PELLETS

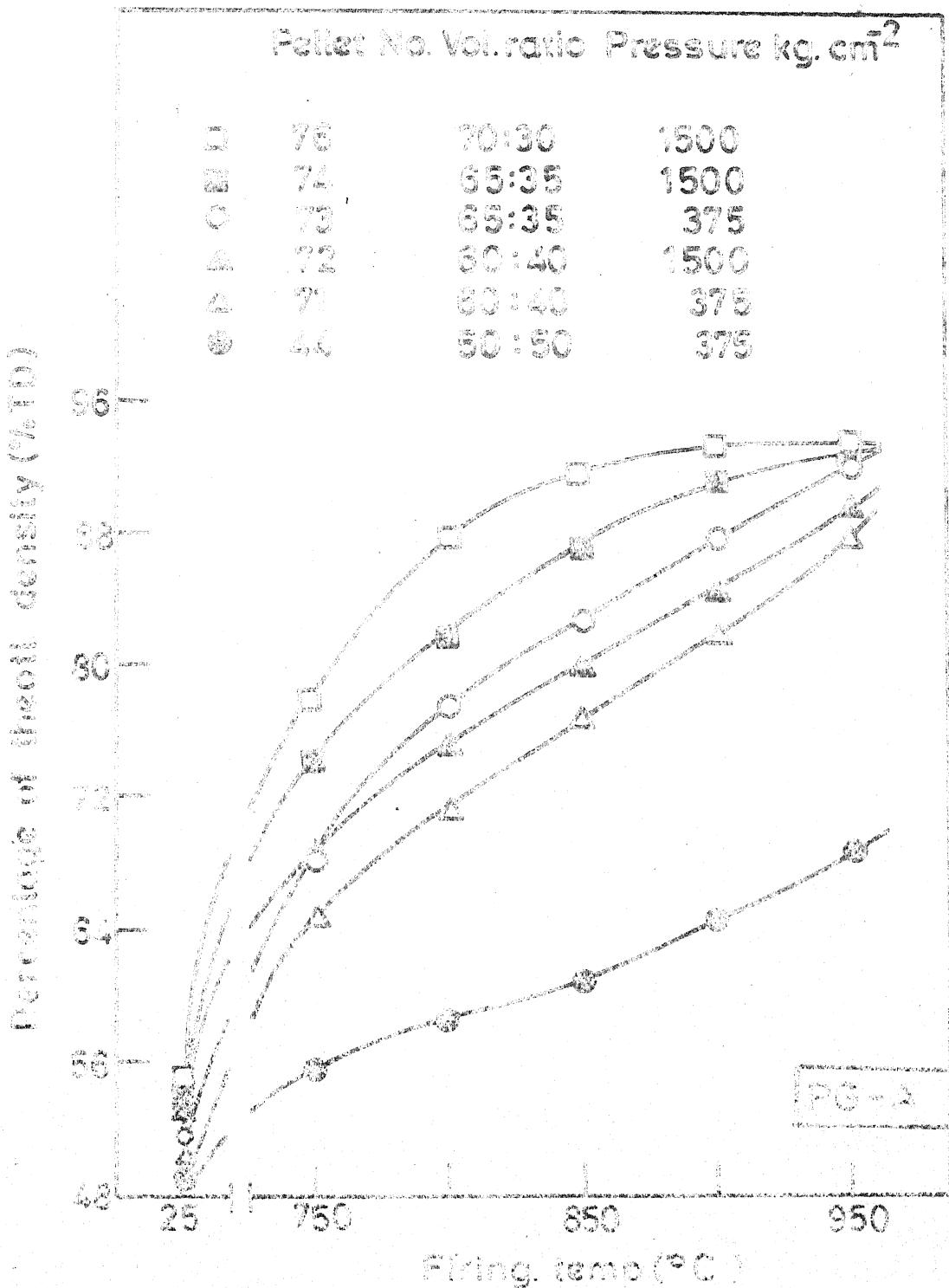


FIG 5.8 PERCENTAGE OF THEORETICAL DENSITY AS A FUNCTION OF FIRING TEMPERATURE FOR PYREX ALUMINA (ach) PELLETS

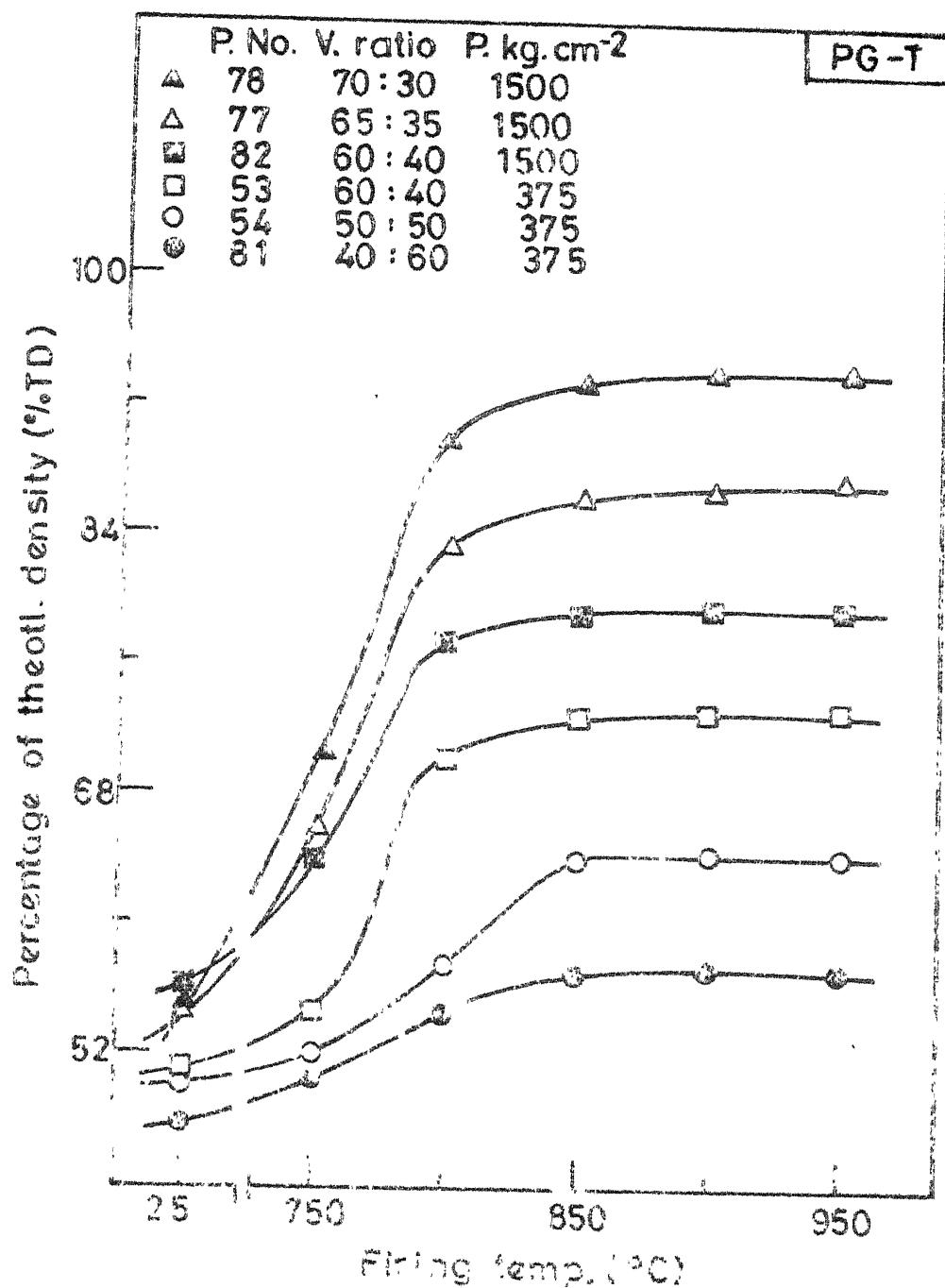


FIG 5.9 PERCENTAGE OF THEORETICAL DENSITY AS A FUNCTION OF FIRING TEMPERATURE FOR PYREX-THERMIT SLAG (40P) PELLETS

Fig. 5.8 shows that unlike Quartz, in Alumina-Pyrex glass system, the sensitivity to bond content as well as pressure is not so marked, specially if the bond content exceeds 65% volume and firing temperatures are around more than 900°C. In this case, densification of the order of 94% of theoretical density can be reached at 900°C.

In case of Thermit Slag in figure 5.9, the results are similar to those of Quartz in figure 5.7. Here again densification of 94% of theoretical density could be attained using 70% volume of bond and 1500 kg cm^{-2} moulding pressure at temperatures of 850°C.

5.5 Effect of Filler Fineness

Although it has been claimed in literature that, finer the size of the filler particles, better are the results, it should be appreciated that grinding is a highly energy intensive unit operation, specially for hard refractory materials such as Alumina and in size ranges of interest i.e. less than 40 microns. In order to confirm whether very fine grinding is really necessary, the best compositions obtained in the previous set of experiments (i.e. 70% bond and 1500 kg cm^{-2} moulding pressure) were repeated using fillers ground for 10 and 20 hours. The results are shown in figures 5.10, 5.11, 5.12 for Quartz, Alumina and Thermit Slag respectively. Surprisingly, coarser powders obtained by 10 hours grinding gave in all three cases

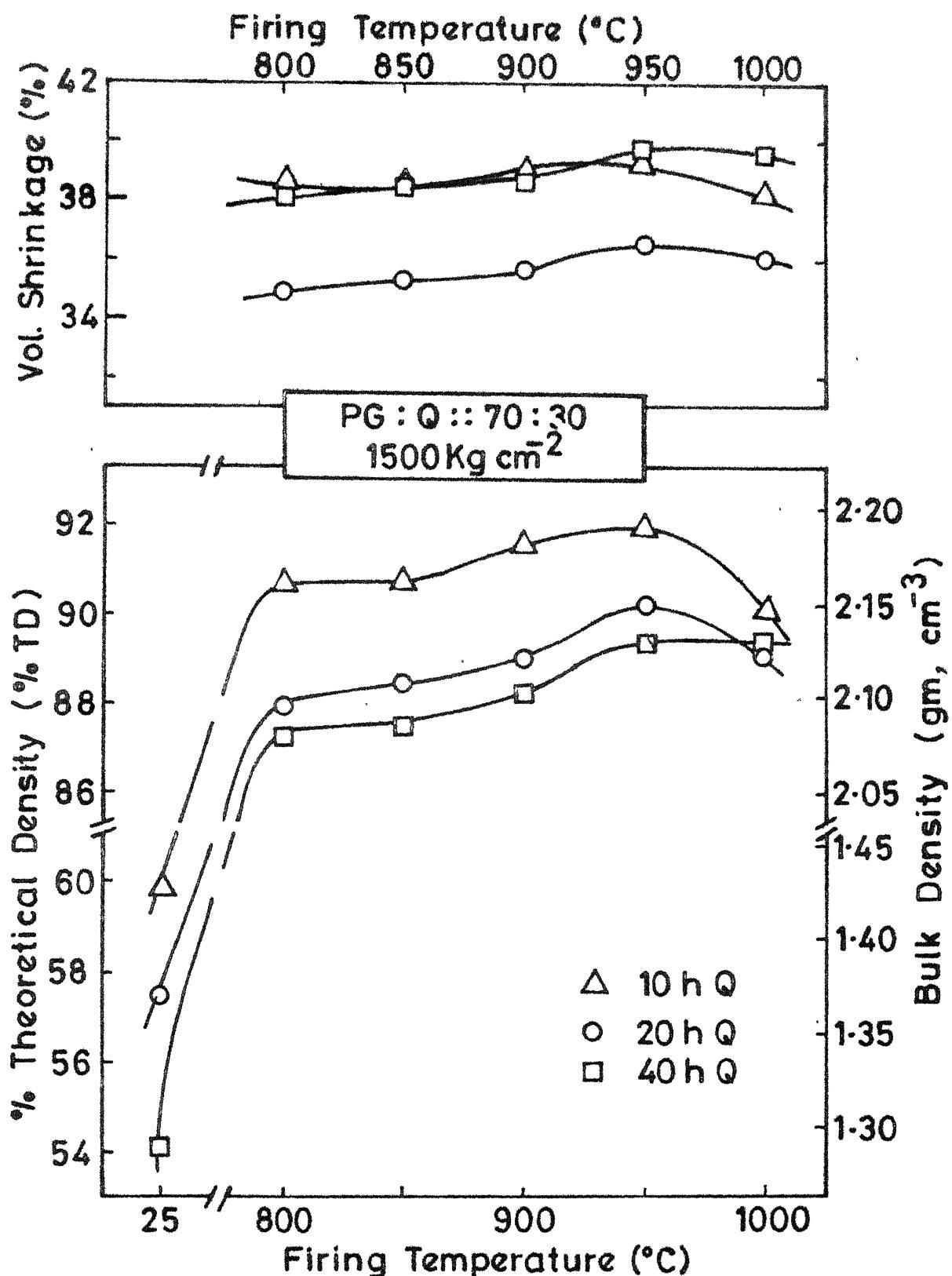


FIG. 5.10. SHRINKAGE, % TD, BULK DENSITY OF PYREX-QUARTZ PELLETS AT DIFFERENT FIRING TEMPERATURES.

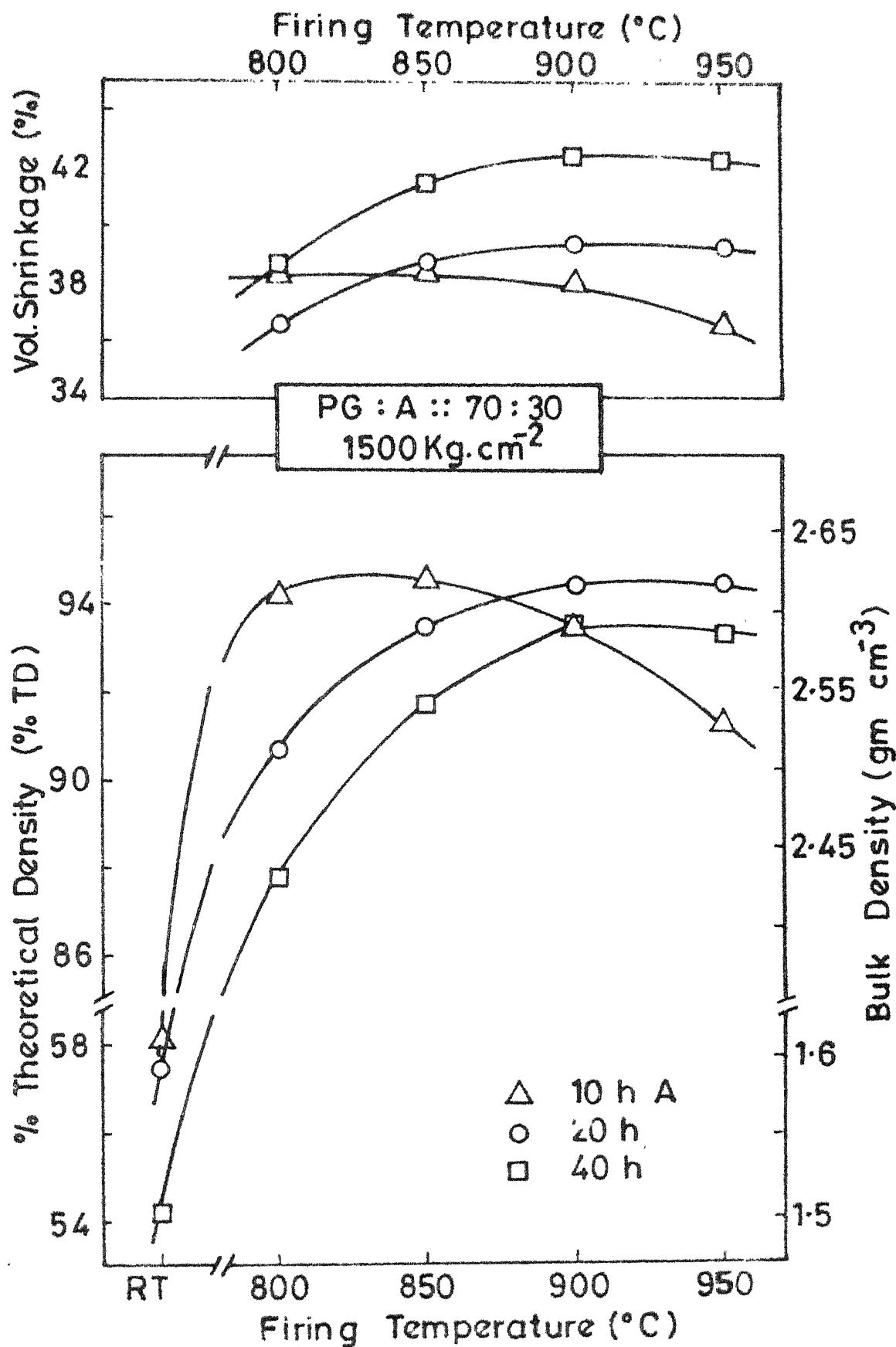


FIG. 5.11. SHRINKAGE, % TD, BULK DENSITY OF PYREX-ALUMINA PELLETS AT DIFFERENT FIRING TEMPERATURES

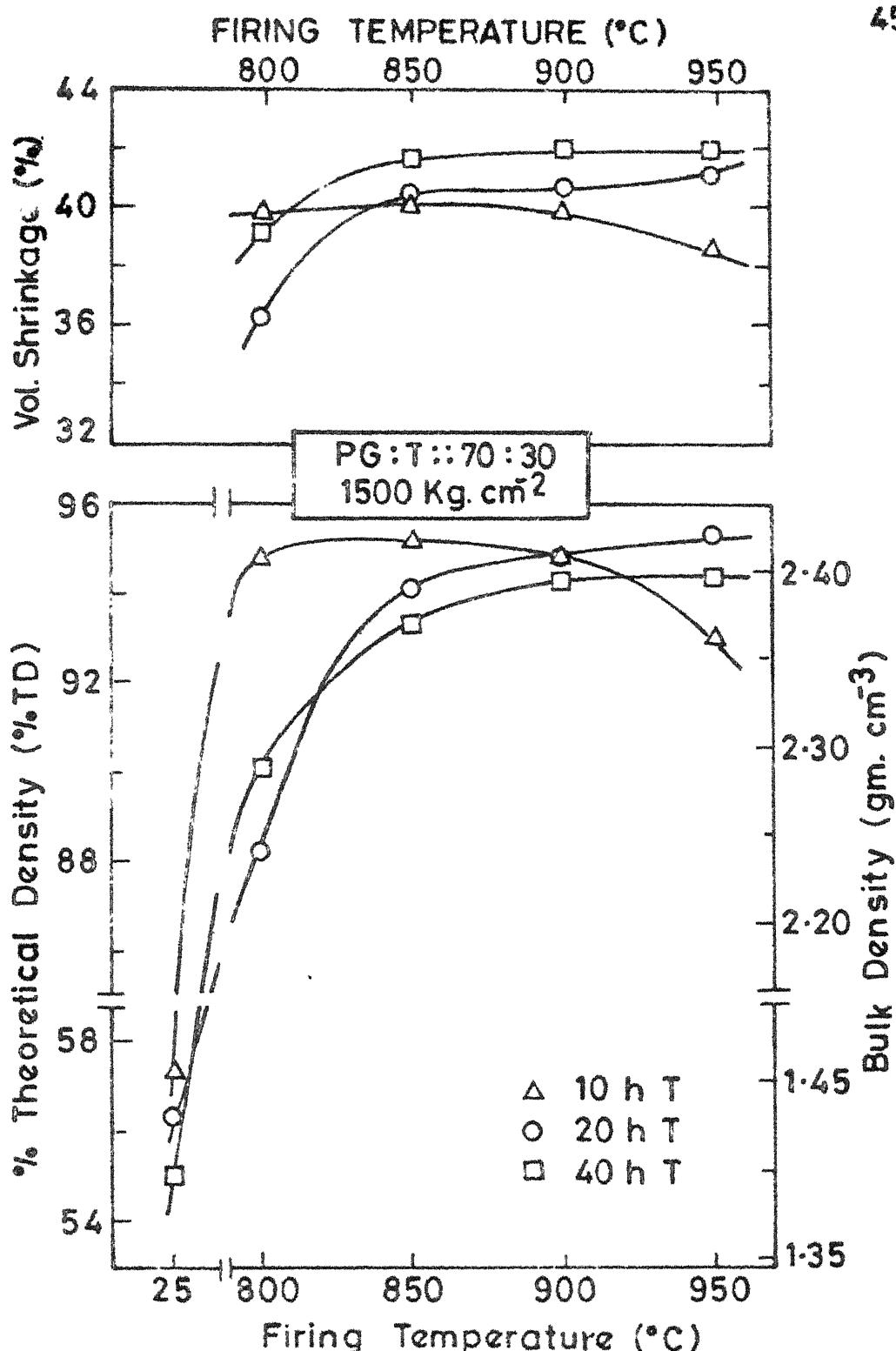


FIG. 5.12. SHRINKAGE, % TD, BULK DENSITY OF PYREX-THERMIT SLAG PELLETS AT DIFFERENT FIRING TEMPERATURES.

as high a densification as given by 40 hours ground powders and not only that, in all cases, the maximum density attained by the coarse powders was at 100 to 150°C lower firing temperatures. It should be however noted that as shown latter, the strength of the bodies were not as high as the bodies with finer fillers.

5.6 Samples for Strength Test

For Quartz, 3 best bodies, and for Alumina and Thermit Slag, 4 best bodies each were selected from the previous set of experiments. For the measurement of the tensile strength by the Brazilian Test, larger samples of 25.4 mm diameter were pressed under 1500 kg cm^{-2} and fired to the same temperature as that of smaller pellets which gave the maximum densification. The fired samples were tested for bulk density by water displacement method, percentage of theoretical density and shrinkage. Water absorption measurements showed that they were mostly in the range of 0.2 to 0.3%, and in no case exceeded 0.5%. The results are shown in tables 5.1, 5.2 and 5.3 for Quartz, Alumina and Thermit Slag respectively.

The strength results for 70% vol bond, are plotted in fig. 5.13. It will be noted that even though the percentage of theoretical density (% TD) was almost constant for Alumina bodies and tended to increase some what for the rest, as the fineness of the filler increased, the maximum strength for all three bodies was located at 20 h grinding time. The effect of

filler fineness on strength is only marginal for the Thermit Slag bodies and most pronounced in the Alumina bodies. It is possible to conclude that there is an optimum particle size in order to realize the best properties and not that finer the filler size, better the properties.

Table 5.1 : Data for Quartz bodies

Code	G	F	E
Quartz, % vol	30(10h)	30(20h)	30(40h)
Pyrex glass, % vol	70	70	70
Firing Temperature °C	840	840	960
Shrinkage, % vol	40.85	42.02	43.16
Bulk density, gm cm ⁻³	2.247	2.253	2.290
% Theoretical density	94.28	94.54	96.50
Tensile strength, kg cm ⁻²	149	173	154

Table 5.2 . Data for Alumina bodies

Code	I	H	A	C
Alumina % vol	30(10h)	30(20h)	30(40h)	35(40h)
Pyrex Glass % vol	70	70	70	65
Firing Temperature °C	825	930	930	930
Shrinkage % vol	38.67	41.54	41.38	41.71
Bulk density gm cm ⁻³	2.601	2.571	2.616	2.692
% Theoretical density	93.87	92.67	94.46	94.23
Tensile strength kg cm ⁻²	145	210	145	136

Table 5.3 : Data for Thermit Slag bodies

Code	K	J	B	D
Thermit Slag % vol	30(10h)	30(20h)	30(40h)	35(40h)
Pyrex Glass % vol	70	70	70	65
Firing Temperature °C	830	935	935	930
Shrinkage % vol	41.66	43.03	43.82	40.75
Bulk density gm cm ⁻³	2.333	2.392	2.472	2.350
% Theoretical density	91.81	94.11	97.43	90.74
Tensile strength kg cm ⁻²	89	92	86	105

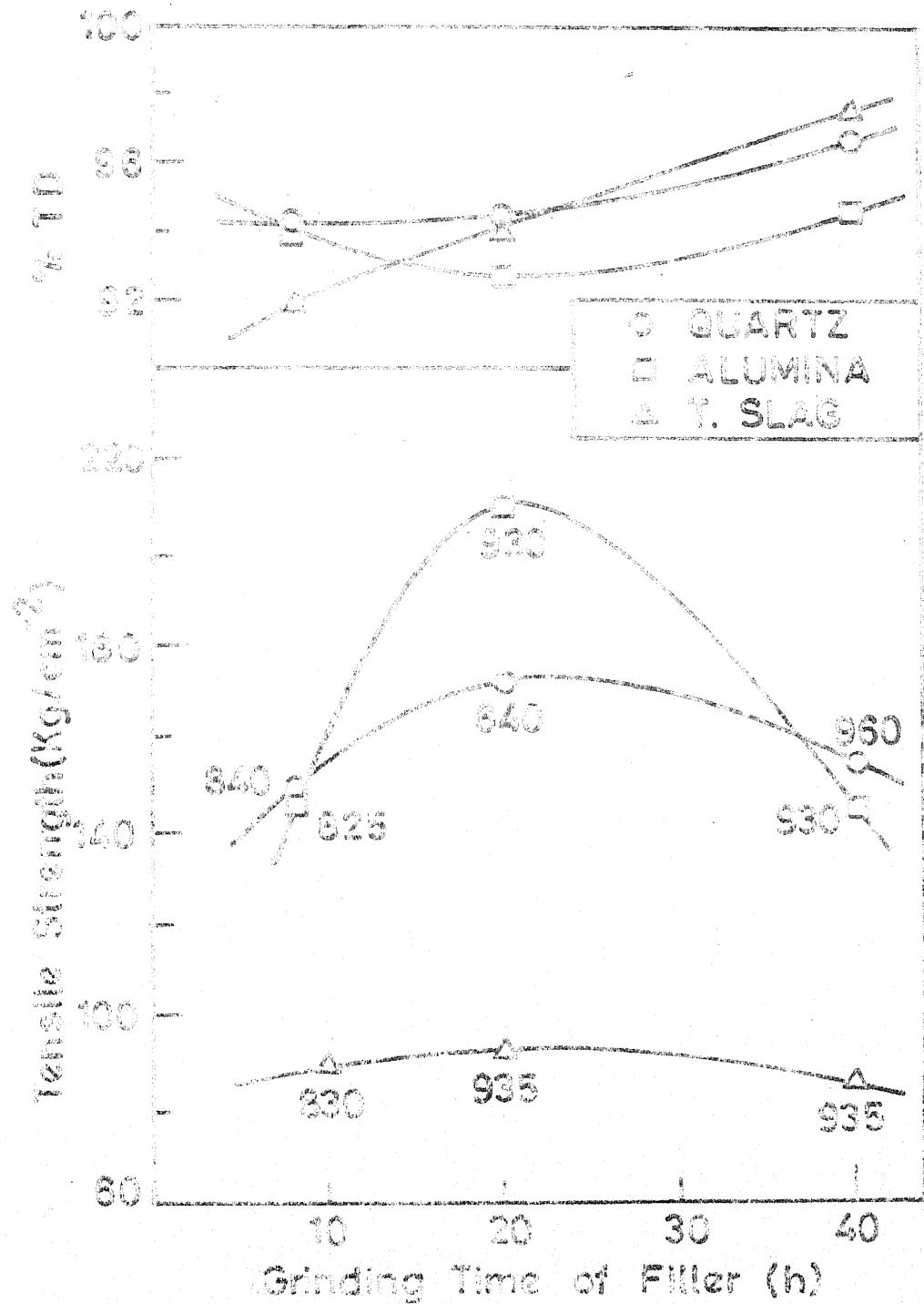


FIG. 5-13. TENSILE STRENGTH AND PERCENTAGE TD AS A FUNCTION OF EXTENT OF GRINDING THE FILLER COMPONENT. FIGURES DENOTE FIRING TEMPERATURE.

CHAPTER-VI

DISCUSSIONS

Of the three glasses tested, Pyrex glass seems to be by far the best for all the three fillers. The reason for its superiority perhaps lies in the fact that its viscosity does not drop with temperature as drastically as in the case of Window glass at least. For E-glass data was not available to permit a similar explanation. This is not to ignore the roll of other factors claimed, for example wetting of filler particles by molten phase, surface tension and its variation with temperature, and possible dissolution of filler, even to a small extent, in the glass. All these factors would differ depending on the nature of the glass.

As shown in figures 5.7 to 5.9, the compaction pressure has an important effect on the ultimate densification reached. This is understandable when one examines the percentage of theoretical densities of green pieces at room temperature. When pressed under 1500 kg cm^{-2} , the green densities tend to go up by approximately 5 to 10% above the densities obtained with 375 kg cm^{-2} pressures. Very roughly, the same increment persists in the densities of the fired samples except for the case of Alumina. In fact Weigmann used isostatic pressing in order to approach 100% theoretical densities. Moreover

there is a definite relationship between the bond content and the moulding pressure. To some extent at least one can be adjusted to compensate for the other as far as the extent of densification is concerned at a given firing temperature.

The firing shrinkages were quite high, of the order of 35 to 40% by volume. This is more or less in agreement with the porosity in the green compact which ranges from 40 to 45% volume. The difference is due to the fact that 100% of theoretical density was not reached. The diametric shrinkages turned out to be 92% to 98% of the shrinkage in height as shown in Appendix III; for pellets of 70% bond content. This could be attributed to the effect of gravity on the pellets which were laid down flat in the furnace. This hypothesis is in confirmity with the fact that when less percentage of bond was used, the two shrinkages tended to be either equal or even their ratio got inverted.

A major outcome of the present investigation seems to be that contrary to the reports in the literature, the fineness of the filler particles must be closely controlled around an optimal size rather than strive for as fine size as possible. If this result can be confirmed by subsequent work, then one must conclude that the cost in grinding the fillers as well as the energy consumed in firing can be reduced still further in these kind of ceramic bodies. This is because, coarser particles reached their maximum densities at lower temperatures than very fine particles.

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CHAPTER-VII

CONCLUSIONS

The following are the principal conclusions arising from this work.

1. Glass bonded ceramic bodies of about 95% of theoretical densities can be made at relatively low firing temperatures of 800-950°C, with Pyrex glass matrix and Alumina, Quartz and Thermit slag fillers.

2. The soak time is about two hours while complete firing cycle required is around 8 to 10 hours, which means a drastic reduction in the firing time as compared to conventional firing practices.

3. For maximum strength, there seems to be an optimal particle size of the filler and not that finer the filler, higher the strength. Moreover coarse particles tend to reach their highest densification at lower temperatures.

4. It is possible to fabricate ceramic bodies having different porosity levels, by decreasing the glassy bond content or firing temperature or moulding pressure for those applications where complete densification is not required.

5. Although three filler materials were studied in this work, it is expected that other ceramic refractory materials like Zirconia, Silicon Carbide, etc. can also be bonded in a similar manner.

6. Reproducibility of result is very good.

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APPENDIX-I

CHARACTERISATION OF RAW MATERIALS

A.I.1 True Density (Specific gravity)

True density was determined by pyknometer method.

Powdered material was used and care was taken to remove all the air bubbles from the powders while specific gravity was determined. Water was used as displacement liquid.

Procedure :

Volume of specific gravity bottle	=	50 cm ³
Mass of the Bottle	=	W gm
Mass of (Bottle + Material)	=	W ₁ gm
Mass of (Bottle + Material + Water)	=	W ₂ gm
Mass of (Bottle + Water)	=	W ₃ gm

$$\text{True density of material} = \frac{(W_1 - W) (W_3 - W)}{(W_3 - W - W_2) 50}$$

Table A.I.1 True density (gm cm⁻³) values for raw materials

Material	Trial No.				Chosen value
	I	III	III	IV	
Window glass	2.493	2.510	2.472	2.473	2.472
E glass	2.545	2.504	2.556	2.555	2.555
Pyrex glass	2.312	2.321	2.252	2.252	2.252
Alumina	4.006	4.016	3.973	3.981	3.980
Quartz	2.671	2.689	2.679	2.681	2.680
Thermit slag	3.220	3.248	3.217	3.212	3.214

A.I.2 Fineness of raw materials (Blaine's Specific Surface Area)

The fineness of the raw materials was assessed by their specific surface areas. The latter was determined by using Blaines apparatus as described in section 4.3.3. All the experiments were conducted at the same room temperature so as to take the viscosity of air constant. Portland cement was used to standardise the apparatus.

Calibration

Height of the packed bed	=	1.536 cm
Diameter of the packed bed	=	1.25 cms
Volume of the packed bed	=	1.855 cm ³
Porosity of the packed bed while standardising (e _s)	=	0.5
Time taken to draw a fixed volume of air to pass through the packed bed while standardising (T _s)	=	31 sec
True density of Portland cement (ρ _s)	=	3.15
Specific surface area of Portland cement	=	2250 cm ² gm ⁻¹

The formula used for computing the specific surface area is

$$S = S_s \sqrt{\frac{s(1 - e_s)}{(1 - e)}} \cdot \sqrt{\frac{e^3}{e_s^3}} \cdot \sqrt{\frac{T}{T_s}}$$

The various symbols are explained in section 4.3.3.

Table A.I.2 Blaine's Specific surface area values for raw materials

Material	ρ gm cm^{-3}	e	T sec	S $\text{cm}^2 \text{ gm}^{-1}$
Thermit Slag				
ground for 40 hrs	3.214	0.60	666.56	21943
ground for 20 hrs	3.214	0.60	647.22	21622
ground for 10 hrs	3.214	0.60	408.44	17177
as received	3.214	0.40	69.22	4135
Alumina				
ground for 40 hrs	3.980	0.55	269.22	6727
ground for 20 hrs	3.980	0.55	127.40	4628
ground for 10 hrs	3.930	0.50	59.33	2464
Quartz				
ground for 40 hrs	2.68	0.55	321.22	9055
ground for 20 hrs	2.68	0.50	194.66	6626
ground for 10 hrs	2.68	0.50	139.45	5609
as received	2.68	0.45	8.00	1042
Window glass				
ground for 25 hrs	2.472	0.50	249.66	8137
Pyrex glass				
ground for 25 hrs	2.252	0.50	231.44	8599
E-glass				
ground for 25 hrs	2.555	0.50	193.11	6923

APPENDIX-II

DATA OF PRELIMINARY EXPERIMENTS

Softening temperatures of standard cones made up of Quartz and Window glass were determined. The Full tip, Half bend, Initial tip temperatures of the cones were as follows.

Material and Vol. Ratio	Tipping temperatures °C		
	Start	Half bend	Full tip
WG; 10 hr Q			
75:25	708	772	790
60:40	735	816	908
50:50	935	1250	1383
WG; 20 hr Q			
75:25	660	727	783
60:40	725	863	917
50:50	908	-	-
WG; 40 hr Q			
75:25	730	769	778
60:40	760	881	934
50:50	881	1150	-

APPENDIX-III

EXPERIMENTAL MEASUREMENTS OF BULK DENSITY,
PERCENTAGE THEORETICAL DENSITY AND SHRINKAGE
OF SMALL PELLETS FIRED AT VARIOUS TEMPERATURES

Abbreviations Used

Mat	Material	
Vol R	Volume Ratio	
Wt R	Weight Ratio	
TD	Theoretical Density	
Comp Pr	Compaction Pressure used in pellet making	kg cm ⁻²
PG	Pyrex glass	
EG	E-glass	
WG	Window glass	
A	Fused Alumina	
Q	Quartz	
T	Thermit Slag	
Q 40 h	Quartz ground for 40 h	
FT	Firing temperature	°C
H	Height	cm
D	Diameter	cm
V	Volume	cm ³
BD	Bulk density	gm cm ⁻³

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 31	25	-	-	-	1.248	48.83
Mat =WG; Q 40h	700	7.34	7.87	21.37	1.586	62.07
Vol R=60:40	750	11.92	11.62	31.22	1.811	70.88
Wt R =0.580:0.419	800	9.17	9.02	24.78	1.657	64.85
TD =2.555	850	5.50	7.95	19.90	1.556	60.90
Comp Pr = 375	900	5.11	5.35	14.93	1.469	57.50
	950	0.66	4.20	8.78	1.369	53.58
	1000	-7.21	1.38	-4.29	1.192	46.65
PELLET No. 32	25	-	-	-	1.257	48.80
Mat = WG; Q 40h	700	2.97	2.98	8.73	1.376	53.42
Vol R= 50:50	750	8.27	9.24	24.47	1.661	64.48
Wt R = 0.48:0.52	800	9.95	11.46	29.46	1.777	68.98
TD = 2.576	850	10.59	11.69	30.33	1.800	69.88
Comp Pr = 375	900	11.63	12.91	33.01	1.871	72.63
	950	12.92	14.05	35.70	1.948	75.62
	1000	12.01	13.98	34.93	1.923	74.65
PELLET No. 33	25	-	-	-	1.120	46.23
Mat = PG; Q 40h	700	4.13	4.32	12.16	1.365	56.34
Vol R= 60:40	750	8.84	8.79	24.14	1.576	65.04
Wt R = 0.557:0.443	800	9.20	9.02	24.82	1.590	65.62
TD = 2.423	850	9.43	9.39	25.69	1.608	66.36
Comp Pr = 375	900	10.14	10.08	27.32	1.643	67.81
	950	11.67	11.44	30.69	1.723	71.11
	1000	13.76	13.64	35.51	1.843	76.06
PELLET No. 34	25	-	-	-	1.218	49.38
Mat = PG; Q 40h	700	1.57	1.67	4.86	1.279	51.87
Vol R= 50:50	750	5.20	5.00	14.49	1.420	57.58
Wt R = 0.457:0.543	800	5.80	5.76	16.34	1.450	58.80
TD = 2.466	850	6.29	6.36	17.84	1.475	59.81
Comp Pr = 375	900	7.38	7.12	20.14	1.516	61.48
	950	8.46	8.71	23.76	1.587	64.36
	1000	10.12	10.68	28.36	1.688	68.45

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 35	25	-	-	-	1.276	48.97
Mat = EG; Q 40h	700	0.00	-0.15	-0.27	1.272	48.83
Vol R= 60:40	750	0.50	-0.08	0.37	1.275	48.94
Wt R = 0.589:0.411	800	1.87	2.05	5.86	1.347	51.71
TD = 2.605	850	6.37	7.06	19.12	1.566	60.12
Comp Pr = 375	900	9.86	9.10	25.53	1.697	65.14
	950	10.11	9.56	26.53	1.719	65.99
	1000	10.49	9.79	27.17	1.733	66.53
PELLET No. 36	25	-	-	-	1.269	48.47
Mat = EG; Q 40h	700	-0.25	-0.46	-1.11	1.254	47.90
Vol R= 50:50	750	-0.13	-0.30	-0.74	1.257	48.01
Wt R = 0.488:0.512	800	0.63	0.76	2.12	1.293	49.39
TD = 2.618	850	2.89	3.11	8.85	1.389	53.03
Comp Pr = 375	900	5.41	5.00	14.65	1.479	56.49
	950	5.41	5.16	14.93	1.481	56.57
	1000	5.66	5.39	15.58	1.490	56.91
PELLET No. 41	25	-	-	-	1.519	49.37
Mat = WG; A 40h	700	4.18	4.06	11.95	1.653	53.73
Vol R= 60:40	750	9.23	8.72	24.39	1.911	62.11
Wt R = 0.482:0.518	800	11.21	10.82	29.62	2.050	66.64
TD = 3.076	850	12.09	11.02	30.44	2.076	67.48
Comp Pr = 375	900	11.76	11.13	30.38	2.073	67.40
	950	11.65	11.02	30.11	2.065	67.14
	1000	11.76	11.09	30.31	2.072	67.34
PELLET No. 42	25	-	-	-	1.564	48.47
Mat = WG; A 40h	700	0.71	0.92	2.45	1.604	49.72
Vol R= 50:50	750	3.53	3.44	9.98	1.725	53.47
Wt R = 0.383:0.617	800	5.88	5.43	15.76	1.841	57.07
TD = 3.226	850	6.12	5.66	16.46	1.857	57.55
Comp Pr = 375	900	6.24	5.58	16.39	1.856	57.53
	950	6.12	5.58	16.29	1.852	57.40
	1000	6.35	5.62	16.64	1.858	57.59

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 43	25	-	-	-	1.459	49.57
Mat = PG; A 40h	700	3.52	4.09	11.29	1.644	55.86
Vol R= 60:40	750	8.81	8.79	24.03	1.919	65.20
Wt R = 0.459:0.541	800	11.23	11.41	30.32	2.089	71.00
TD = 2.943	850	13.66	13.57	35.48	2.257	76.69
Comp Pr = 375	900	16.85	15.92	41.13	2.476	84.14
	950	18.50	17.89	45.00	2.646	89.92
	1000	19.16	18.35	46.13	2.702	91.80
PELLET No. 44	25	-	-	-	1.521	48.78
Mat = PG;A40h	700	1.37	1.60	4.53	1.592	51.08
Vol R= 50:50	750	4.11	4.33	12.25	1.729	55.47
Wt R = 0.361:0.639	800	5.94	6.08	17.11	1.827	58.60
TD = 3.117	850	7.08	7.37	20.30	1.900	60.99
Comp Pr = 375	900	8.90	9.12	24.83	2.014	64.63
	950	10.96	10.94	29.36	2.144	68.78
	1000	12.10	11.85	31.71	2.217	71.12
PELLET No. 45	25	-	-	-	1.566	50.14
Mat = EG;A40h	700	0.23	0.00	0.32	1.570	50.26
Vol R= 60:40	750	0.23	0.15	0.49	1.570	50.25
Wt R = 0.491:0.509	800	0.69	0.76	2.36	1.600	51.05
TD = 3.124	850	3.00	3.19	9.14	1.706	54.62
Comp Pr = 375	900	7.85	6.99	20.33	1.946	62.68
	950	11.32	9.72	27.78	2.144	68.62
PELLET No. 46	25	-	-	-	1.646	50.38
Mat = EG;A40 h	700	0	0.23	0.53	1.653	50.61
Vol R= 50:50	750	0	0.30	0.53	1.654	50.63
Wt R = 0.391:0.609	800	0.24	0.38	1.06	1.654	50.62
TD = 3.267	850	0.96	1.06	3.17	1.683	51.53
Comp Pr = 375	900	3.36	2.89	8.80	1.788	54.74
	950	5.03	4.40	13.20	1.878	57.49

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 51	25	-	-	-	1.350	48.76
Mat = WG; T 40h	700	1.58	2.29	6.02	1.439	51.95
Vol R= 60:40	750	2.70	3.28	8.86	1.469	53.04
Wt R = 0.536:0.464	800	6.30	6.34	17.72	1.612	58.22
TD = 2.769	850	6.76	6.56	18.56	1.610	58.20
Comp Pr = 375	900	6.98	6.64	18.89	1.618	58.43
	950	6.98	6.64	18.89	1.616	58.36
PELLET No. 53	25	-	-	-	1.344	50.99
Mat = PG; T 40 h	700	1.27	1.29	3.90	1.397	53.00
Vol R= 60:40	750	2.65	2.96	8.42	1.444	54.78
Wt R = 0.513:0.487	800	11.04	11.01	29.64	1.850	70.20
TD = 2.636	850	12.52	12.08	32.45	1.920	72.86
Comp Pr = 375	900	12.74	12.23	32.76	1.930	73.21
	950	12.95	12.31	33.07	1.935	73.42
PELLET No. 54	25	-	-	-	1.364	49.94
Mat = PG; T 40 h	700	1.32	1.29	3.88	1.420	51.95
Vol R= 50:50	750	1.98	2.05	5.99	1.421	51.98
Wt R = 0.412:0.588	800	5.73	5.70	16.18	1.574	57.60
TD = 2.733	850	9.69	9.19	25.57	1.755	64.21
Comp Pr = 375	900	9.91	9.35	26.05	1.765	64.59
	950	9.91	9.35	26.05	1.761	64.43
PELLET No. 55	25	-	-	-	1.502	53.28
Mat = EG; T 40 h	700	0.23	0.23	0.70	1.512	53.65
Vol R= 60:40	750	0.47	0.46	1.38	1.509	53.53
Wt R = 0.544:0.456	800	1.41	1.75	4.84	1.545	54.82
TD = 2.819	850	1.88	2.89	7.60	1.577	55.92
Comp Pr = 375	900	4.69	5.25	14.51	1.698	60.19
	950	4.93	5.34	15.03	1.706	60.53
PELLET No. 52	25	-	-	-	1.421	49.98
Mat = WG; 40 h	700	1.43	1.83	5.11	1.496	52.61
Vol R= 50:50	750	2.14	2.38	6.70	1.506	52.98
Wt R = 0.435:0.565	800	3.33	3.36	9.70	1.535	53.98
TD = 2.843	850	3.80	3.74	10.93	1.543	54.26
Comp Pr = 375	900	3.80	3.67	10.76	1.540	54.17
	950	3.80	3.82	11.11	1.545	54.35

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 56	25	-	-	-	1.548	53.67
Mat = EG; T 40 h	700	0.24	0.23	0.53	1.559	54.05
Vol R= 50:50	750	0.48	0.53	1.43	1.556	53.95
Wt R = 0.443:0.557	800	1.45	1.75	4.81	1.590	55.12
TD = 2.884	850	2.17	2.74	7.31	1.619	56.13
Comp Pr = 375	900	3.62	3.96	11.05	1.677	58.15
	950	3.62	3.81	10.70	1.664	57.69
PELLET No. 71	25	-	-	-	1.450	49.31
Mat = PG; A 40h	750	8.64	8.51	23.52	1.896	64.42
Vol R= 60:40	800	11.88	11.47	30.84	2.092	71.10
Wt R = 0.459:0.541	850	14.04	13.60	35.77	2.252	76.52
TD = 2.943	900	16.20	15.35	39.90	2.404	81.67
Comp Pr = 375	950	18.36	17.25	44.04	2.585	87.82
PELLET No. 72	25	-	-	-	1.619	55.06
Mat = PG; A 40h	750	7.31	7.03	19.89	2.023	68.74
Vol R= 60:40	800	10.24	9.76	27.00	2.213	75.10
Wt R = 0.459:0.541	850	12.44	11.50	31.44	2.353	79.93
TD = 2.943	900	14.15	13.01	35.17	2.487	84.50
Comp Pr = 1500	950	16.10	14.52	38.72	2.632	89.43
PELLET No. 73	25	-	-	-	1.396	49.79
Mat = PG; A 40h	750	10.06	10.17	37.36	1.960	68.61
Vol R= 65:35	800	13.70	13.59	35.53	2.205	77.19
Wt R = 0.512:0.488	850	15.85	15.49	39.94	2.359	82.57
TD = 2.857	900	17.56	17.16	43.40	2.506	87.72
Comp Pr = 375	950	19.06	18.38	46.07	2.629	92.02
PELLET No. 74	25	-	-	-	1.557	54.49
Mat = PG; A 40h	750	9.93	9.66	26.68	2.114	73.99
Vol R= 65:35	800	13.32	12.45	33.55	2.331	81.59
Wt R = 0.512:0.488	850	15.35	14.26	37.81	2.489	87.12
TD = 2.859	900	16.70	15.40	40.43	2.598	90.91
Comp Pr = 1500	950	17.16	15.92	41.41	2.645	92.57

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 75	25	-	-	-	1.357	48.99
Mat = PG; A 40h	750	13.70	13.59	35.59	2.106	76.03
Vol R= 70:30	800	17.38	16.92	43.09	2.380	85.95
Wt R = 0.569:0.431	850	19.22	18.45	46.40	2.524	91.11
TD = 2.770	900	19.94	18.98	47.45	2.577	93.02
Comp Pr = 375	950	19.84	18.98	47.30	2.573	92.90
PELLET No. 76	25	-	-	-	1.499	54.13
Mat = PG; A 40h	750	11.67	11.31	30.46	2.158	77.91
Vol R= 70:30	800	15.64	14.71	38.56	2.430	87.73
Wt R = 0.569:0.431	850	17.18	15.76	41.31	2.537	91.61
ID = 2.770	900	17.62	16.37	42.42	2.588	93.43
Comp Pr = 1500	950	17.62	16.29	42.26	2.583	93.27
PELLET No. 77	25	-	-	-	1.417	54.71
Mat = PG; T 40h	750	5.91	5.97	16.66	1.703	65.76
Vol R= 65:35	800	13.95	12.84	34.54	2.154	83.19
Wt R = 0.565:0.435	850	15.13	13.82	36.94	2.231	86.18
TD = 2.589	900	15.13	13.97	37.29	2.245	86.70
Comp Pr = 1500	950	15.37	14.12	37.63	2.259	87.26
PELLET No. 78	25	-	-	-	1.398	55.01
Mat = PG; T 40h	750	7.72	7.92	21.73	1.787	70.32
Vol R= 70:30	800	15.69	15.09	39.22	2.289	90.07
W5 R = 0.62:0.38	850	17.10	16.00	41.60	2.372	93.35
TD = 2.541	900	17.33	16.23	41.94	2.395	94.24
Comp Pr = 1500	950	17.56	16.06	41.94	2.396	94.28
PELLET No. 81	25	-	-	-	1.348	47.66
Mat = PG; T 40h	750	1.89	2.13	6.09	1.435	50.71
Vol R= 40:60	800	4.49	4.71	13.22	1.543	54.53
Wt R = 0.318:0.682	850	6.38	6.31	17.74	1.641	57.03
TD = 2.83	900	6.62	6.38	18.08	1.625	57.40
Comp Pr = 375	950	6.62	6.46	18.26	1.620	57.27

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 82	25	-	-	-	1.479	56.09
Mat = PG; T 40h	750	3.91	4.61	12.66	1.691	64.15
Vol R = 60:40	800	11.25	9.91	27.99	2.040	77.39
Wt R = 0.512:0.488	850	11.98	10.59	29.59	2.086	79.13
TD = 2.636	900	11.98	10.74	29.76	2.093	79.40
Comp Pr = 1500	950	11.98	10.82	29.95	2.091	79.34
PELLET No. 83	25	-	-	-	1.333	55.01
Mat = PG; Q 40h	750	6.08	6.70	18.25	1.630	67.28
Vol R = 60:40	800	7.84	9.33	24.19	1.757	72.53
Wt R = 0.588:0.412	850	10.00	9.78	26.73	1.818	75.02
TD = 2.423	900	10.78	10.38	28.29	1.858	76.70
Comp Pr = 1500	950	12.16	11.59	31.26	1.939	80.03
	1000	13.72	13.54	35.50	2.065	85.21
PELLET No. 84	25	-	-	-	1.299	54.09
Mat = PG; Q 40 h	750	10.45	11.10	29.23	1.836	76.44
Vol R = 65:35	800	11.64	11.93	31.52	1.892	78.76
Wt R = 0.609:0.391	850	11.64	12.01	31.52	1.895	78.90
TD = 2.402	900	11.83	12.16	31.95	1.904	79.26
Comp Pr = 1500	950	12.43	12.76	33.38	1.941	80.81
	1000	12.62	13.74	34.95	1.990	82.85
PELLET No. 85	25	-	-	-	1.289	54.07
Mat = PG; Q 40 h	750	13.88	14.34	36.79	2.039	85.57
Vol R = 70:30	800	14.69	14.87	38.10	2.079	87.25
Wt R = 0.662:0.337	850	14.69	15.02	38.39	2.087	87.56
TD = 2.383	900	15.09	15.17	38.83	2.102	88.19
Comp Pr = 1500	950	15.49	15.55	39.71	2.130	89.40
	1000	15.29	15.62	39.71	2.130	89.40
PELLET No. 86	25	-	-	-	1.455	57.28
Mat = PG; T 10h	750	11.75	12.79	32.87	2.168	85.33
Vol R = 70:30	800	15.57	15.52	39.84	2.409	94.78
Wt R = 0.62:0.38	850	15.85	15.67	40.24	2.418	95.17
TD = 2.541	900	15.57	15.59	39.84	2.410	94.83
Comp Pr = 1500	950	14.75	15.14	38.65	2.361	92.91

Details	FT	% Shrinkage			BD	% TD
		H	D	V		
PELLET No. 87	25	-	-	-	1.609	58.09
Mat = PG; A 10h	800	15.63	14.49	38.22	2.608	94.15
Vol R = 70:30	850	15.87	14.49	38.39	2.616	94.43
Wt R = 0.569:0.431	900	15.38	14.34	37.87	2.586	93.36
TD = 2.770	950	14.18	13.96	36.47	2.528	91.25
Comp Pr = 1500						
PELLET No. 88	25	-	-	-	1.425	59.86
Mat = PG; Q 10h	800	15.21	14.76	38.35	2.159	90.59
Vol R = 70:30	850	15.21	14.76	38.35	2.161	90.67
Wt R = 0.662:0.337	900	15.63	14.91	38.95	2.179	91.44
TD = 2.383	950	15.63	15.14	39.25	2.191	91.92
Comp Pr = 1500	1000	14.58	14.91	38.20	2.155	90.04
PELLET No. 89	25	-	-	-	1.430	56.29
Mat = PG; T 20h	800	14.19	13.77	36.33	2.241	88.20
Vol R = 70:30	850	16.70	15.36	40.33	2.396	94.11
Wt R = 0.620:0.380	900	16.93	15.50	40.66	2.406	94.70
TD = 2.541	950	17.16	15.73	41.16	2.423	95.37
Comp Pr = 1500						
PELLET No. 90	25	-	-	-	1.592	57.46
Mat = PG; A 20h	750	11.37	14.54	35.27	2.459	88.79
Vol R = 70:30	800	14.69	13.79	36.64	2.510	90.63
Wt R = 0.569:0.431	850	15.64	14.62	38.53	2.588	93.44
TD = 2.770	900	16.11	14.85	39.21	2.617	94.47
Comp Pr = 1500	950	16.11	14.85	39.21	2.617	94.47
PELLET No. 91	25	-	-	-	1.369	57.44
Mat = PG; Q 20h	750	10.23	10.77	28.57	1.915	80.38
Vol R = 70:30	800	13.18	13.34	34.81	2.094	87.89
Wt R = 0.662:0.337	850	13.41	13.49	35.30	2.107	88.43
TD = 2.383	900	13.64	13.64	35.63	2.120	88.96
Comp Pr = 1500	950	14.09	14.02	36.45	2.150	90.22
	1000	13.41	13.94	35.96	2.127	89.25

APPENDIX-IV

EXPERIMENTAL MEASUREMENTS OF BULK DENSITY, PERCENTAGE THEORETICAL DENSITY, SHRINKAGE, WATER ABSORPTION AND TENSILE STRENGTH OF BIG SAMPLES

Abbreviations used other than those in Appendix III

T.S Tensile Strength

* Reading not considered

† Volume of the standard samples was found by water displacement method

Water Ab Water Absorption

Details	No	% Shrinkage			BD	% TD	% Water Ab	T.S
		H	D	V				
CODE A								
Mat = PG; A 40h	A1	16.88	16.03	41.42	2.605	94.06	0.272	
Vol R = 70:30	A2	16.87	16.22	41.02	2.616	94.45	0.276	
Wt R = 0.569:0.431	A3	17.26	16.22	41.23	2.637	95.21	0.191	134*
TD = 2.770	A4	17.18	15.99	41.39	2.606	94.11	0.320	
Comp Pr = 1500	A5	16.84	15.87	41.29	2.65	94.05	0.346	
F.T = 930	A6	17.81	15.99	41.93	2.627	94.85	0.255	145
Average Values		17.14	16.05	41.38	2.616	94.46	0.277	145
CODE B								
Mat = PG; T 40h	B1	17.32	17.09	43.37	2.498	98.30	0.224	
Vol R = 70:30	B2	17.51	17.44	43.76	2.497	98.26	0.279	
Wt R = 0.62:0.33	B3	18.16	17.40	44.27	2.470	97.20	0.366	86
TD = 2.541	B4	18.09	17.37	43.91	2.458	96.74	0.302	79*
Comp Pr = 1500	B5	17.96	17.33	43.81	2.457	96.71	0.374	
F.T = 935	B6	-	-	-	2.463	96.93	0.284	
Average Values		17.84	17.33	43.82	2.474	97.43	0.288	86
CODE C								
Mat = PG; A 40h	C1	17.05	15.83	40.81	2.690	94.16	0.191	136
Vol R = 65:35	C2	17.34	16.06	42.22	2.683	93.90	0.276	110*
Wt R = 0.512:0.488	C3	13.84	16.18	42.39	2.703	94.62	0.249	
TD = 2.857								
Comp Pr = 1500								
F.T = 930								
Average Values		17.74	16.02	41.71	2.692	94.23	0.239	136
CODE D								
Mat = PG; T 40h	D1	16.96	15.53	41.12	2.353	90.87	0.356	
Vol R = 65:35	D2	16.68	15.37	40.18	2.327	89.87	0.293	
Wt R = 0.565:0.435	D3	16.45	15.25	40.28	2.351	90.82	0.307	
TD = 2.589	D4	16.54	15.29	40.35	2.340	90.39	0.285	
Comp Pr = 1500	D5	16.56	15.25	40.54	2.354	90.92	0.197	105
F.T = 930	D6	17.12	15.72	41.37	2.352	90.84	0.312	71*
Average Values	D7	17.25	15.64	41.39	2.369	91.49	0.193	
		16.79	15.44	40.75	2.350	90.74	0.278	105

Details	No	% Shrinkage			BD	%TD	Percent water Ab.	T.
		H	D	V				
CODE E	E1	17.62	16.72	44.13	2.295	96.32	0.292	
Mat = PG; Q 40h	E2	12.98*	16.37	42.69	2.276	96.53	0.344	
Vol R = 70:30	E3	17.28	16.49	42.63	2.287	95.96	0.322	10
Wt R = 0.662:0.337	E4	17.35	16.87	42.83	2.288	96.01	0.189	15
TD = 2.383	E5	17.68	16.49	43.52	2.305	96.72	0.319	
Comp Pr = 1500								
E.T = 960								
Average values		17.48	16.59	43.16	2.290	96.50	0.293	15
CODE F								
Mat = PG; Q 20 h	F1	16.74	16.17	41.54	2.241	94.06	0.549	
Vol R = 70:30	F2	17.25	16.40	42.63	2.275	95.49	0.306	17
Wt R = 0.662:0.337	F3	16.65	15.90	41.43	2.233	93.70	0.444	11
TD = 2.383	F5	17.30	16.52	42.46	2.276	95.52	0.440	
Comp Pr = 1500	F6	17.16	16.29	42.05	2.339	93.95	0.436	
F.T. = 840								
Average values		17.02	16.26	42.02	2.253	94.54	0.435	17
CODE G								
Mat = PG; Q 10h	G1	17.16	15.78	41.85	2.248	94.33	0.364	
Vol R = 70:30	G2	15.76	15.51	40.76	2.241	94.04	0.347	
Wt R = 0.662:0.337	G3	16.20	15.74	35.21	2.052*	86.12*	0.336	14
TD = 2.383	G4	16.40	15.70	40.79	2.246	94.24	0.644	11
Comp Pr = 1500	G5	16.26	15.70	40.87	2.259	94.80	0.351	
F.T. = 840								
Average Values		16.36	15.69	40.85	2.247	94.28	0.408	14
CODE H								
Mat = PG; A 20h	H1	16.08	15.78	40.46	2.555	92.25	0.52	21
Vol R = 70:30	H2	22.51*	15.62	45.08	2.579	93.12	0.35	
Wt R = 0.569:0.431	H3	18.94	15.23	41.90	2.552	92.14	0.36	
TD = 2.770	H4	13.24	15.93	38.70	2.571	92.82	0.31	
Comp Pr = 1500	H5	9.6*	15.47	35.72*	2.576	93.01	0.27	10
F.T. = 930								
Average values		16.09	15.61	41.54	2.571	92.67	0.36	21

Details	No	% Shrinkage			BD	%TD	Percent water		T..
		H	D	V			Ab	Ab	
CODE I									
Mat = PG; A 10h	I1	15.13	14.60	-	2.598	93.80	0.37		
Vol R = 70:30	I2	14.96	14.44	38.66	2.603	93.98	0.28		14
Wt R = 0.569:0.431	I3	15.38	14.68	38.38	2.589	93.48	0.27		
TD = 2.770	I4	15.44	14.87	30.96	2.619	94.55	0.29		14
Comp Pr = 1500	I5	11.41*	13.21*	35.24*	2.591	93.53	0.34		
F.T. = 825									
Average values		15.24	14.65	30.67	2.601	93.87	0.31		14
CODE J									
Mat = PG; T 20 h	J1	17.86	16.48	42.42	2.388	93.96	0.37		
Vol R = 70:30	J2	20.95*	16.97	44.84	2.389	94.05	0.32		
Wt R = 0.62:0.38	J3	17.86	16.52	42.59	2.396	94.28	0.31		2
TD = 2.541	J4	18.12	16.48	42.68	2.395	94.25	0.34		9
Comp Pr = 1500	J5	17.10	16.40	42.61	2.394	94.23	0.38		
F.T. = 935									
Average values		17.73	16.57	43.03	2.392	94.11	0.34		92
CODE K									
Mat = PG; T 10 h	K1	17.60	16.08	41.78	2.351	92.52	0.33		
Vol R = 70:30	K2	12.94*	15.31	41.43	2.332	91.79	0.41		29
Wt R = 0.62:0.38	K3	17.67	16.23	42.32	2.338	92.02	0.35		52
TD = 2.541	K4	17.64	15.81	41.44	2.312	90.90	0.73		
Comp Pr = 1500	K5	17.39	15.81	41.35	2.331	91.73	0.45		
F.T. = 830									
Average values		17.58	15.98	41.66	2.333	91.81	0.45		89